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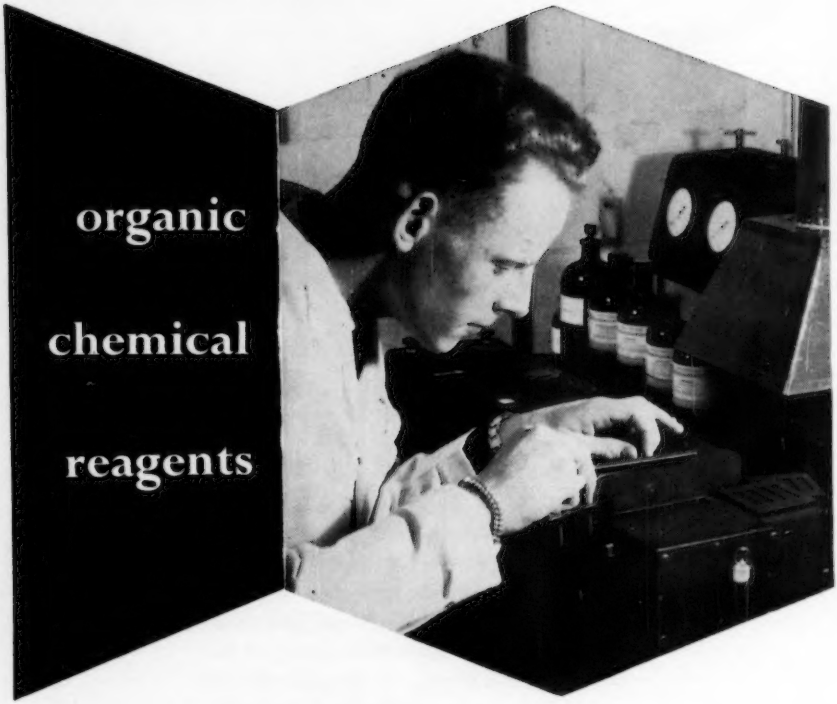

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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

General reviews of progress, reagents and methods of general application.

2825. **Applied reviews.** *Anal. Chem.*, 1959, **31** (4, Part II).—This number presents a summary of progress during the previous two years in applying analytical techniques and instrumentation in fifteen major fields. The following contributions are included. **Air pollution,** K. Kay, 633–645. **Automatic operations in analytical chemistry,** G. D. Patterson, jun., 646–655. **Clinical chemistry,** G. R. Kingsley, 656–674. **Coatings,** M. H. Swann, M. L. Adams and G. G. Esposito, 674–678. **Essential oils and related products,** E. Guenther, K. Kulka and J. A. Rogers, 679–687. **Fertilisers,** J. A. Brabson, 688–691. **Food,** K. Morgareidge, 691–696. **Solid and gaseous fuels,** W. H. Ode, 697–706. **Ferrous metallurgy,** H. F. Beeghly, 706–712. **Non-ferrous metallurgy,** G. H. Farrah and M. L. Moss, 713–723. **Pesticides,** W. E. Westlake, 724–729. **Petroleum,** R. L. LeTourneau, 730–749. **Pharmaceuticals and related drugs,** C. R. Rehm, S. C. Slack and W. J. Mader, 749–766. **Natural and synthetic rubbers,** M. Tryon and F. J. Linnig, 767–776. **Water analysis,** L. L. Thatcher and R. T. Kiser, 776–789.

2826. **Evaluation and error in physical analyses.** H. Kienitz (Ammoniaklab., Badisch. Anilin- & Sodafabrik A.-G., Ludwigshafen, Rhein, Germany). *Z. anal. Chem.*, 1958, **164** (1), 80–99.—The quant. evaluation of data and checking of errors in physicochemical analyses, with special reference to mass spectrometry, infra-red and ultra-violet absorption spectra, are discussed. B. B. BAUMINGER

2827. **Combination of physical methods of measurement in chemical analysis.** H. Günzler (Ammoniaklab., Badisch. Anilin- & Sodafabrik A.-G., Ludwigshafen, Rhein, Germany). *Z. anal. Chem.*, 1958, **164** (1), 49–56.—The advantages of combining ultra-violet, infra-red and Raman spectroscopy, mass spectrometry and gas chromatography in the analysis of olefins are discussed.

B. B. BAUMINGER

2828. **Ion exchange and chromatography in analytical chemistry.** ASTM, S.T.P. No. 195, 1958, 57 pp.—A report of a symposium including the following contributions. **Ion exchange in analytical chemistry,** R. Kunin, 3–10. **Ion exchange in analytical and radiochemistry,** J. E. Hudgens, jun., 11–25. **Metal separations by anion exchange,** K. A. Kraus and F. Nelson, 27–57.

2829. **Organic reagents for inorganic analysis. IV. Syntheses of new phenylfluorone derivatives, and their reactions with metal ions.** Hirotoshi Sano (Dept. of Chem., Univ. of Tokyo, Hongo). *Bull. Chem. Soc. Japan*, 1958, **31** (8), 974–980 (in English). —Phenylfluorone (**I**) derivatives were prepared by condensation of *m*- or *p*-nitro- or *o*-sulpho-benzaldehyde with 1:2:4-triacetoxybenzene. The *m*-nitrophenyl derivative was found to be superior to the others and to unsubstituted **I** in stability, sensitivity to Ti^{4+}, Ge^{4+} and Zr^{4+}, and in low optical absorbance when made up as a blank solution.

S.C.I. ABSTR.

2830. **Rubeanic acid [dithio-oxamide] and its derivatives as colorimetric reagents. IV. NN′-Dibenzyl- and NN′-diphenyl-rubeanic acids.** J. Xavier and Priyadaranjan Rây Indian Ass. for the Cultivation of Sci., Jadavpur, Calcutta). *J. Indian Chem. Soc.*, 1958, **35** (10), 725–732.—NN′-Dibenzyl-rubeanic acid gives metal complexes similar to those of the parent compound, but with lower solubilities in common organic solvents. NN′-Diphenyl-rubeanic acid, though highly coloured, can be used with even greater advantage than rubeanic acid itself for the colorimetric determination of Cu, Ni, Co and Pd. The reagent is more sensitive than its parent compound or its alkyl analogues. The behaviour of thio-oxanilide towards various metal cations is studied. This reagent is much less effective for analytical purposes.

I. JONES

2831. **Studies on oxine and its derivatives. The sensitivity and selectivity of some 7-substituted 8-hydroxyquinoline-5-sulphonic acids and some 2-substituted 8-hydroxyquinoline-4-carboxylic acids towards certain metals.** R. G. W. Hollingshead (B.D.H. Ltd., Chemicals Div., Poole, Dorset, England). *Anal. Chim. Acta*, 1958, **19** (5), 447–457. —Directions are given for the prep. of 8-hydroxy-7-morpholinomethylquinoline-5-sulphonic acid, 8-hydroxy-7-piperidinomethylquinoline-5-sulphonic acid, 2-(3:4-dimethoxyphenyl)-8-hydroxyquinoline-4-carboxylic acid, 2-*n*-hexyl-8-hydroxyquinoline-4-carboxylic acid (**I**) and 2-(1-ethylpropyl)-8-hydroxyquinoline-4-carboxylic acid. The behaviour of these reagents with Cu^{2+}, Ni^{2+}, Al^{3+}, Zn^{2+}, Mg^{2+}, Ga^{3+}, In^{3+}, Tl^{3+}, Y^{3+}, Cd^{2+}, Co^{2+} and Fe^{3+} has been studied in buffer soln. of pH 5·3, 8·35 and 13·1. Only **I** gives a ppt. with Al^{3+}. The other ions studied give, in general, colour reactions of low sensitivity (mainly yellow or green) with all reagents tested. An exception to this is Tl^{3+} which react with **I** at pH 5·3 and 8·35 to give a rose-pink colour stable for 15 min. None of the derivatives shows any marked analytical advantage over the parent compound. T. R. ANDREW

2832. **Systematic study of insoluble substances. I.** I. K. Taimni and I. S. Ahuja (Chem. Lab., Univ. of Allahabad, India). *Anal. Chim. Acta*, 1958, **19** (5), 492–495.—'Insoluble' substances met with in qual. analysis are defined as 80% resistant to 5 successive evaporations to dryness of 0·5 g with 15 ml of HNO_3 followed by 15 ml of HCl when the HNO_3 vol. falls to 5 ml. The following

were insoluble according to this test—nitroprussides of Co, Ni and Cu; ferricyanides of Ni and Co; ignited chromites of Mg, Zn, Cd, Mn, Co, Cu, Ni, Be, Ce, U and Zr; ignited aluminates of Cu, Co, Zn, Mn and Ni; ignited arsenates of Ti, Zr and Cr; ignited chromium phosphate and freshly prepared manganic metaphosphate. T. R. ANDREW

2833. **Ammonium salt of N-2-naphthylmalonamic acid as a reagent for the quantitative estimation of metals.** M. L. Bhambhani and H. D. Mathur (Chem. Dept., St. John's College, Agra, India). *Agra Univ. J. Res., Sci.*, 1958, **7** (1), 93–96.—Zinc, cobalt, manganese and copper can be quant. determined with considerable accuracy by pptn. with N-2-naphthylmalonamic acid from aq. soln., and drying and weighing the ppt. Zinc and manganese can be separated by washing the ppt. with 25% ethanol, when the Mn compound dissolves. N. E.

2834. **Nucleation in analytical chemistry.** D. H. Klein and L. Gordon (Dept. of Chem. and Chem. Engng, Case Inst. of Technol., Cleveland, Ohio, U.S.A.). *Talanta*, 1958, **1** (4), 334–343.—The formation of a ppt. involves two steps, first nucleation, consisting in the formation within a supersaturated soln. of the first particles of ppt. capable of spontaneous growth, and secondly the growth of these particles as ions are deposited on them. The two current theoretical approaches to nucleation are discussed, with 22 references. It is concluded that, as the nature of nucleation is further elucidated, the basic procedures used in analytical chemistry to favour the growth of crystalline ppt. by controlling supersaturation will need revision and extension to include nucleation phenomena, since during the nucleation period the number of particles and the final size are fixed, and nucleation determines the surface area on which pptn. can take place, and therefore establishes the tempo of the pptn. R. E. ESSERY

2835. **Precipitations in chemical analysis and the solubility-product principle. III. Competition for solvent molecules.** S. Lewin (S.-W. Essex Tech. Coll., London). *Lab. Practice*, 1958, **7** (11), 637–643.—The limits of the region of concn. in which the solubility-product theory is strictly valid, and the need for taking into account solvent transport are discussed, with the aid of a mathematical treatment of the competition for solvation molecules during the co-saturation solubility of two very sol. substances or electrolytes. This treatment of solvent transport, combined with the theory of free-energy changes under equilibrium conditions, is then applied to co-solubility data for NaCl and ethanol in H_2O, KCl (or KI) - KBr, KCl - KNO_3 and NaCl - KCl in H_2O. W. J. BAKER

2836. **New indicator for argentimetric determinations.** N. F. Dobrovol'skiĭ. *Trudy Khar'kovsk. Med. Inst.*, 1957 (1958), **15** (36), 397–400; *Ref. Zhur., Khim., Biol. Khim.*, 1959, (6), Abstr. No. 6330.—The indicator suggested is 1:5-di(anilino)-pentamethine chloride (**I**). *Procedure*—To 0·08 *N* $AgNO_3$ (20 ml) add a 0·08% soln. of **I** in 50% ethanol (0·6 to 1·8 ml) and titrate with 0·08 *N* NaCl. Before the end of the titration add ethanol (to give a concn. $\geq$ 30%). The suspension turns pink at the end-point. In the titration of Cl^- with $AgNO_3$, the end-point is reached when the soln. and suspension turn yellow owing to desorption of **I**. The determination of I^- is similar to that of Cl^-. The error in the determination of Cl^- and I^- is 0·1 to 0·35%. K. R. C.

2837. **New sensitive indicator for semi-micro chelometric titrations.** R. H. Maier (Dept. of Agric. Chem. and Soils, Univ. of Arizona, Tucson, U.S.A.). *Nature*, 1959, **183**, 461–462.—The reagent 3-hydroxy-4-*o*-hydroxyphenylazonaphth-2′:4′-xylidide termed "magon" introduced for spectrophotometric determination of Mg (Mann and Yoe, *Anal. Abstr.*, 1957, **4**, 2109) is used as indicator in titration of microgram amounts of Mg with EDTA. For the range 0 to 100 μg of Mg, the recommended procedure includes addition to the sample soln. of Clark and Lubs' buffer pH 9·0 (10 ml), abs. methanol (15 ml), and magon soln. (0·1 mg per ml in methanol, freshly prepared) (10 ml). This soln. is adjusted if necessary to pH 10·5 and titrated with 0·005 *N* EDTA from yellow - red to blue - violet. Methanol in the titration soln. should be in a concn. of 30 to 80%; the effect of Fe, Cu, etc. (< 50 μg), may be eliminated by adding 1 to 3 mg of KCN before the magon. H. F. W. KIRKPATRICK

2838. **Lead as indicator in amperometric titrations with sodium diethyldithiocarbamate solution.** Yu. I. Usatenko and F. M. Tulyupa (F. É. Dzerzhinskiĭ Dniepropetrovsk Chem. Tech. Inst.). *Zavod. Lab.*, 1958, **24** (11), 1327–1331.—Soln. of Na diethyldithiocarbamate (NaR) are oxidised on the rotating platinum micro-electrode giving waves starting at + 0·4 and + 0·8 V *vs.* the S.C.E. When Pb is titrated amperometrically at an applied potential of + 0·8 V, the first additions of the reagent produce no immediate ppt. and the diffusion current increases until pptn. starts when the current decreases. It is supposed that the soluble PbR^+ are formed first. In the presence of other metals, PbR^+ form earlier than the dithiocarbamates of Fe, Mn, Ni and Cd, and later than those of Cu and Hg. Lead can serve as an indicator for amperometric titrations of Cu and Hg in the presence of many other metals. To the slightly acid soln. containing 0·2 to 4·0 mg of Hg or 0·06 to 2·0 mg of Cu are added 2 to 5 ml of 0·1 *M* $Pb(NO_3)_2$ (the molar ratio of Pb to Ni must be < 3), 0·5 g of K tartrate, 1 g of Na acetate, 5 to 10 ml of saturated NH_4Cl soln., and water to give a vol. of 40 to 50 ml. The pH should be between 4 and 7. A platinum electrode is introduced and connection is made to a S.C.E. Titration is carried out with a 0·01 *M* soln. of the reagent at an applied potential of 0·8 to 0·9 V. G. S. SMITH

2839. **Calculation of titration curves and titration errors.** R. Wyatt (Tech. High School, Eindhoven, Netherlands). *Chem. Weekbl.*, 1958, **54** (46), 631–635.—A method is presented for the calculation of the slope of the titration curve at the end-point. The precision of the titration follows directly from this. G. BURGER

2840. **Photometric titrations.** J. B. Headridge (Chem. Dept., Sheffield Univ., England). *Talanta*, 1958, **1** (4), 293–304.—The three main types of photometric titrations are described, namely, those without added indicator, those with more than enough indicator to complex the substance being titrated or post-end-point additions of titrant, and those in which the indicator is sufficient to complex only a part of these. Possible sources of error are mentioned, and a literature survey is

given, with 119 references from 1954 to 1957, covering titrations in the above three classes, review articles and theoretical papers, coulometric titrations with photometric end-points, and titrations in the u.v. region of the spectrum.
R. E. ESSERY

2841. **Contribution to the problem of the membrane electrode. The determination of end-point with the aid of membrane potential. I. Precipitation titrations with silver salts.** J. Bersier, P. Bersier, W. Trueb and F. Hügli (Abt. f. Chem., Tech., Winterthur, Switzerland). *Helv. Chim. Acta*, 1959, **42** (1), 55–60.—A membrane of aluminium oxide, deposited by anodic oxidation of aluminium, is used as an indicator in pptn. titrations with silver salts. By correct choice of electrolytes, a large change of membrane potential occurs at the end-point of the titration. A. R. ROGERS

2842. **Symposium on spectrochemical analysis for trace elements.** ASTM, S.T.P. No. 221, 1958, 79 pp.—This publication contains the following contributions—**Emission spectrometric determination of oxygen in metals,** V. A. Fassel, W. A. Gordon and R. W. Tabeling, 3–22. **Spectrographic determination of trace elements in metals,** J. A. Norris, 23–38. **Trace analysis by means of the graphite spark,** J. M. Morris and F. X. Pink, 39–46. **Spectrochemical analysis for trace elements in geological materials,** K. J. Murata, 67–79.

2843. **Spectroscopic methods as aids for solving techno-chemical problems.** H. Luther (Inst. f. Chem. Technol., Tech. Hochschule, Braunschweig, Germany). *Z. anal. Chem.*, 1958, **164** (1), 109–120.—Measuring evaporation equilibria, observing dissolution and diffusion processes and determining courses of reaction, etc., have been successfully carried out by i.r. spectroscopy. The analysis of low mol. wt. org. substances, the determination of coeff. of diffusion of solvents, *i.e.*, plasticisers (phthalates, sebacates, etc.) in poly(vinyl chloride), the course of reaction of polymerisation of propenyl propyl ether with $AlCl_3$ and the oxidisability of mineral-oil fractions are quoted as examples.
B. B. BAUMINGER

2.—INORGANIC ANALYSIS

General, determination of elements (arranged in the order of the Periodic Table), analysis of minerals and inorganic industrial products.

2844. **Analysis of the lighter elements by total reflection of their characteristic X-ray wavelengths.** A. Franks and R. F. Braybrook (Nat. Phys. Lab., Teddington, Mddx., England). *Brit. J. Appl. Phys.*, 1959, **10** (4), 190–191.—A collimated beam of X-rays excited from the sample is allowed to fall at a glancing (adjustable) angle on to a reflector, and the reflected X-rays are detected by means of a windowless photomultiplier. A plot of relative intensity against glancing angle enables the angle of maximum intensity to be determined. This is characteristic for each element. It is suggested that the procedure might be applied to the determination of one light element (Al, Mg or Na) in admixture with heavy elements. It is not advocated for mixtures of light elements. T. R. ANDREW

2845. **Determination of silver, cadmium, indium and tin in silver-base alloys.** D. P. Stricos and J. T. Porter, II (Knolls Atomic Power Lab., Schenectady, N.Y.). *U.S. Atomic Energy Comm.*, Rep. KAPL–M–DPS–1, July 17, 1958, 13 pp.—The sample is dissolved in a mixture of HNO_3 and HBF_4; the Ag and In are determined polarographically in KI soln., and the Sn is determined polarographically in NH_4Cl - HCl. NUCL. SCI. ABSTR.

2846. **Ultra-violet spectrophotometric simultaneous determination of iron, lead and bismuth in hydrochloric acid.** Yuroku Yamamoto (Ishibashi Lab., Kyoto Univ., Japan). *Bull. Inst. Chem. Res. Kyoto Univ.*, 1958, **36** (6), 139–144 (in English).—The absorption spectra of Fe^{III}, Bi and Pb in various concn. of HCl soln. are given. These indicate that it is possible to determine these metals in the presence of each other in 6 *M* HCl soln. Copper, Sb, V and Ti interfere, but it is suggested that these be removed by a procedure described by Merritt (*Anal. Chem.*, 1953, **25**, 572). The absorbance of each metal is measured for a range of concn. from 0 p.p.m. to 15 p.p.m. in 20 ml, at 270 mμ (Pb peak), 327 mμ (Bi peak), and 357 mμ (Fe peak). A simple factor is found relating absorbance to concn. at each wavelength for each metal, and Beer's law applies. Since the Fe spectrum forms a plateau from 270 mμ to 370 mμ, it is necessary to correct the absorbances at 270 mμ and 327 mμ for the amount of Fe found by means of the 357-mμ peak. Similarly, the slight absorption of Bi at 270 mμ must be allowed for. The error claimed is 3·2% for Fe, 2·2% for Pb, and 1·2% for Bi. H. M.

2847. **Argentimetric titrations with redox indication. Determination of halides, cyanides, thiocyanates and potassium in the presence of Variamine blue.** L. Erdey, I. Buzás and K. Vigh (Inst. für allg. Chem., Tech. Univ., Budapest, Hungary). *Talanta*, 1958, **1** (4), 377–395 (in German).—The redox properties of Variamine blue are discussed, and potentiometric titration curves are given. At a suitable pH, and in the presence of small amounts of oxidising agents (*e.g.*, Ag^+), a blue oxidation product is formed. The chemistry of this system is discussed. The preparation of the indicator, used as a 1% soln. in 20% acetic acid, is described, and the procedure for the titration of Cl^- (1 to 150 mg) at pH 3·6 (acetate buffer), and of Br^- (0·4 to 400 mg), I^- (0·6 to 600 mg), SCN^- (0·3 to 300 mg) and CN^- (0·1 to 400 mg) at pH 4·65 (acetate buffer) is given. The soln. are treated with NaOH or HNO_3 to a faint pink with phenolphthalein, then just decolorised with 0·1 *N* acetic acid; 10 ml of buffer and 3 drops of indicator are added, and the mixture is titrated with 0·1 *N* $AgNO_3$ to a blue coloration. Near the end-point, the titrant is added dropwise, with shaking after each addition. Silver (5 to 550 mg) can be determined by neutralising the soln. to phenolphthalein as described above, faintly acidifying with acetic acid and titrating with 0·1 or 0·01 *N* KI till the blue is discharged, warming to 40° to 50° near the end-point. For 0·1 *N*, 0·01 *N* and 0·001 *N* soln. the standard deviations are ± 0·04 to 0·07%, ± 0·08 to 0·51% and ± 0·16 to 0·25%, respectively. Iodine and KI can be determined in tincture of iodine (2 ml) in 50% ethanol (10 ml) by titrating the free iodine with ascorbic acid at pH 4·65 until the indicator colour is discharged, then continuing the titration with $AgNO_3$ until the colour re-appears, giving total iodine, KI being obtained by difference. Potassium (10 to 100 mg) is determined by pptn. with sodium tetraphenylboron, washing the ppt. with water containing 0·5 to 1 g of Al_2O_3, dissolving

it in acetone (10 to 20 ml), acidifying with 2 ml of *N* acetic acid, adding 3 drops of indicator, and titrating with $AgNO_3$ soln. to a violet end-point. Notes on the determinations are given. Results for the titration of Cl^- in potable water are in good agreement with those from methods in which $KCrO_4$ is used as indicator. R. E. ESSERY

2848. **Determination of gases in metals.** ASTM, S.T.P. No. 222, 1958, 60 pp.—This contains the following publications—**Two apparatuses for the determination of gases in metals,** D. L. Guernsey and R. H. Franklin, 3–14. **The application of vacuum fusion to gas - metal studies,** A. L. Beach and W. G. Guldnes, 15–24. **Oxygen determination using a platinum bath and capillary trap,** W. G. Smiley, 25–32. **Bromination - carbon reduction method for the determination of oxygen in metals,** M. Codell, G. Norwitz and S. Kallmann, 33–40. **Emission spectrometric determination of oxygen in metals,** V. A. Fassel, W. A. Gordon and R. W. Tabeling, 41–60.

2849. **Use of oscillographic polarography in quantitative analysis. Micro-determination of several metals.** R. Kalvoda (Polarographic Inst., Czechoslovak Acad. Sci.). *Zavod. Lab.*, 1958, **24** (11), 1319–1326.—The method that involves the preliminary formation of amalgams (*cf. Anal. Abstr.*, 1957, **4**, 3553) is described. It is applied to the determination of metals in the concn. range 10^{-5} to 10^{-9} *M*. G. S. SMITH

2850. **Application of paper electrophoresis to the separation of radioactive products found in the Szilard - Chalmers reaction.** J. Jach, H. Kawahara and G. Hardbottle (Brookhaven Nat. Lab., Upton, Long Island, N.Y., U.S.A.). *J. Chromatography*, 1958, **1** (6), 501–507.—Many of the radioactive ionic species formed in typical Szilard - Chalmers reactions (*Nature*, 1934, **134**, 462) can be separated by paper electrophoresis, which can also assist identification. The effectiveness of the technique is demonstrated by separation of the ionic species found in irradiated alkali bromates, KIO_3, KIO_4, As_2O_5, $K_3Co(CN)_6$ and bromoacetic acid. G. P. COOK

2851. **Quantitative determination of fission and nuclear reaction products.** C. E. Crouthamel, R. Heinrich and C. Gatrousis (Argonne Nat. Lab., Lemont, Ill., U.S.A.). *Talanta*, 1958, **1** (4), 396–407.—The analysis of nuclear fuel as practised at the Argonne National Laboratory is described. The depletion of fuel in a nuclear reactor is discussed, and definitions given of "per cent. total atom burn-up" and "per cent. atoms consumed". The paper is largely concerned with the determination of the fraction of fissile atoms consumed by fission in nuclear fuels, but many of the basic methods of calibration and detection are applicable to the analysis of other nuclear reaction products. The discussion covers some of the calibrations and techniques employed, the types of instrumentation applicable and the elucidation of some of the special problems encountered, and includes the direct measurement of fission events by counting and the measurement of heat evolution, fission-product monitoring by means of ^{137}Cs (including the isolation of Cs from burn-up samples by paper chromatography in HF or HNO_3 soln.) and the assay of neutron capture and other nuclear reaction products by radiochemical and mass-spectrographic methods. (22 references.) R. E. ESSERY

2852. **Quantitative analysis by a spectrographic method of the isotopic composition of hydrogen, deuterium and tritium mixtures.** M. N. Oganov and A. R. Striganov. *Spectrochim. Acta*, 1958, **13** (1–2), 139–149.—This is an English translation of an article in Russian that appeared originally in *Atomn. Energiya*, 1957, **2**, 112; *cf. Anal. Abstr.*, 1959, **6**, 30. B. B. BAUMINGER

2853. **Simplified float method for the determination of the deuterium oxide content of water.** P. Baertschi and M. Thürkauf (Phys.-chem. Inst., Univ. Basel). *Helv. Chim. Acta*, 1959, **42** (1), 282–293.—An accuracy of better than 0·01% deuterium-content is easily attained by means of very simple equipment for the purification of the water sample and for the determination of the temperature at which the glass or quartz float sinks or rises in the sample. Numerical tables are given for the calculation of the isotopic composition, and the corrections that have to be applied are discussed. A. R. ROGERS

2854. **Micro-determination of gases in water vapour.** J. Hissel (CEBEDEAU, 2, rue Armand Stévart, Liége, Belgium). *Bull. Centre Belge Étude Document. Eaux*, 1958, (42), 269–275.—The application on the micro scale of a method based on Henry's law is critically studied. After an exposition of the theory, an apparatus and experimental procedure are described for the study of the partition of H (0·5 and 1 ml) between water and argon at 15°. The theoretical partition coefficient is higher than the observed one by 2 to 3% of the amount of H, corresponding to an excess of 1 to 2 μg of H in the water. It is also shown that, contrary to expectation from Henry's law, the volume of the gaseous phase seems to have little influence on its content of H as determined by gas chromatography. A comparison by gas chromatography of two methods of extraction of H from water (into a vacuum, and into an inert gas) shows wide discrepancies, which should not be the case if Henry's law is applicable, and similar discrepancies are shown for O between results by extraction into a vacuum and by a coulometric Winkler method. It is concluded that methods based on Henry's law are unsuitable for the determination of very small amounts of gases in water, probably because other phenomena superimpose their effects on the classical partition of a gas between the two phases. R. E. ESSERY

2855. **Equilibrium moisture content determination at high temperature.** W. A. Wink, F. C. Bobb and J. A. Van den Akker (Inst. Paper Chem., Appleton, Wis., U.S.A.). *TAPPI*, 1958, **41**, 643–646.—The sorption isotherm of a hygroscopic material at high temp. is determined by passing a stream of air at predetermined temp. and humidity through a chamber in which the specimen is held in a shallow Petri dish suspended on a glass rod, which passes through the vertical exhaust tube of the chamber and is attached to a balance beam. Details of procedure and a sketch of the apparatus are given, together with results on the desorption of paper at 93° through a relative v.p. range of 0·979 to 0·202. W. HOLDEN

2856. **Differential spectrophotometric determination of hydrogen peroxide using 1:10-phenanthroline and bathophenanthroline.** R. Bailey and D. F. Boltz (Wayne State Univ., Detroit, Mich., U.S.A.). *Anal. Chem.*, 1959, **31** (1), 117–119.—

The method described is based on the decrease in absorbance of the Fe^{II} - phenanthroline complex in the presence of H_2O_2. For determining 0·1 to 2·5 p.p.m. of H_2O_2, 1:10-phenanthroline is used and for the 0·03 to 0·1 p.p.m. range 4:7-diphenyl-1:10-phenanthroline (bathophenanthroline) is used. A considerable number of ions interfere unless their concn. is controlled. K. A. PROCTOR

2857. **Measurement of impurities in helium. I. The katharometer as a continuous analyser for total impurities.** M. Wilkins and J. D. Wilson (A.E.R.E., Harwell, Berks., England). A.E.R.E. Report C/R 2008, 1959, 21 pp.—Two katharometers have been studied. Both had potentialities for use as continuous monitors for impurities other than H and D in helium. Sensitivities were determined for A, O, N and air and were about 1000 p.p.m. and 5000 p.p.m. for full-scale deflections in the two instruments. If H and D are present, only small corrections have to be made to readings for other impurities. G. J. HUNTER

2858. **Determination of oxygen and hydrogen in helium.** W. R. Marsh (A.E.R.E., Harwell, Berks., England). A.E.R.E. Report C/M 377, 1959, 14 pp.—The galvanic cell of Hersch (*cf.* Mond Nickel Co., Ltd., *Anal. Abstr.*, 1957, **4**, 781) has been applied to the determination of oxygen in helium. By bleeding into the gas stream a known additional amount of oxygen and measuring the residual oxygen after passing over a "Deoxo" catalyst the hydrogen content of the gas may be calculated. Results (14) are quoted for hydrogen in nitrogen at concn. from 14 to 1670 p.p.m., with an average error of 8 p.p.m. T. R. ANDREW

2859. **Isotopic analysis of oxygen in water.** M. Anbar (Isotope Dept. of the Weizmann Inst. of Sci., Rehovoth, Israel). *Intern. J. Appl. Radiation and Isotopes*, 1958, **3** (2), 131–134.—The water (50 to 100 mg) is added to a glass ampoule containing $CoCl_2$ (1 to 3 mg) and Na (10 to 20 mg). The mixture is frozen, melted and de-gassed, and frozen again; bromine vapour (20 ml) is distilled into the ampoule which is then sealed and warmed for 20 to 30 min., after which it may be stored indefinitely before analysis of the O by mass spectrometry. As little as 12·5 mg of water can be analysed. G. J. HUNTER

2860. **Gas - solid chromatographic analysis of fractions from air rectification columns.** S. A. Greene (Aerojet-General Corp., Azusa, Calif., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 480.—The rare gases are obtained from side streams which are bled from various sections of air rectification columns and this paper describes how such mixtures might be monitored and analysed by gas-adsorption chromatography. K. A. PROCTOR

2861. **Quantitative spectral analysis of gas mixtures.** O. Botschkowa, S. Frisch and E. Schreider (Phys. Res. Inst., Univ. Leningrad, USSR). *Spectrochim. Acta*, 1958, **13** (1–2), 50–59 (in German).—A high-frequency discharge with external electrodes is used as the light source. The discharge is stimulated by a high-frequency generator of which a diagram is shown. Analyses of rare gases containing N, H, O and hydrocarbons, of small amounts of Kr in oxygen, of N - CO_2 mixtures, and of three-component mixtures (He, Ne and A) are discussed. By using discharges in narrow capillaries at a pressure of 100 mm, 10^{-4} to 10^{-5} % of the added gas can be determined. Spectral analysis by impulse discharge, resulting in an increase in sensitivity, is also referred to.

B. B. BAUMINGER

2862. **Determination of hydrogen in alkali metals by isotope dilution method.** B. D. Holt (Argonne Nat. Lab., Lemont, Ill., U.S.A.). *Anal. Chem.*, 1959, **31** (1), 51–54.—From 5 to 250 p.p.m. of H in a 2-g sample of sodium or sodium - potassium can be determined to within ± 2 p.p.m. by equilibration with deuterium for 5 min. at 460° in an 8-in. borosilicate-glass tube, which also serves as the gas-bulb for mass-spectrometric analysis. The tube is flamed to decompose condensed hydrides and deuterides before attachment to the mass spectrometer for the recording of outputs of mass numbers 2, 3 and 4. For sodium - potassium, a correction factor should be applied to compensate for the effect of different rates of formation of hydrides and deuterides in the cooler zones of the tube. This effect is negligible for sodium. An analysis can be made in ≃ 45 min. after taking the sample directly from the liquid system.

W. J. BAKER

2863. **Flame-photometric determination of lithium in different organic solvents.** D. D. Vukanović. *Bull. Inst. Nucl. Sci. "Boris Kidrich", Belgrade*, 1958, **8** (148), 43–51 (in English).—The effects of certain physicochemical properties of organic solvents on the flame-photometric determination of LiCl were investigated. The surface tension of the aliphatic alcohols used (22·27 to 27·5 dynes per cm) exerts very little effect on the curve. Increased sensitivity of the determination is obtained with an increase in flame temperature, which is a function of the heat of combustion of the solvent, which in turn is a function of the mol. wt. of the solvent. It is also shown that the rate of flow of the solvent (expressed in ml per min.) is a function of the viscosity. If the effect of the flow rate is eliminated, the intensity of lithium emission increases considerably with an increase in the mol. wt. of the aliphatic alcohols studied. K. R. C.

2864. **Paper-chromatographic separation and determination of lithium, sodium and potassium.** F. Modreanu (Acad. R.P.R., Iasi, Romania). *J. Chromatography*, 1958, **1** (6), 554–555 (in French).—The propionates of Li, Na and K are separated by using the ascending technique and ethanol - water - propionic acid (100:10:1) as the developer. The spots are located by application of an aq. soln. of violuric acid. R_F values are, for K, 0·17; Na, 0·31; and Li, 0·68. G. P. COOK

2865. **Flame photometer for measuring sodium aerosol concentrations in furnace gases.** D. Ounsted (Brit. Coal Utilis. Res. Ass., Randalls Rd., Leatherhead, England). *J. Inst. Fuel*, 1958, **31**, 474–479.—The concn. of sodium salts volatilised from the fuel bed and carried in the furnace gases is determined by drawing the gases, by an electrically driven fan, through a cyclone to precipitate ash particles, and injecting, by means of an air-jet at 10 lb per sq. in., a portion of the clean gases, measured by an orifice plate, into a chamber where it mixes with the air supply to the photometric flame. The photometer readings are calibrated by drawing a simultaneous sample of the cleaned gases through an electrostatic (20 kV) discharge tube at 150°, which completely precipitates the aerosol

so that its sodium content can be recovered and determined. The graph shows a small intercept ($\simeq$ 2 p.p.m.) on the sodium concn. axis which is probably due to slight pptn. of the aerosol in the sampling tube. For concn. $>$ 20 p.p.m., the furnace gases are diluted. Variations in the CO_2 content of the flue gases affect the luminosity due to Na so that a max. error of $\pm$ 5% might occur in normal practice; calibration graphs for flue gases of different CO_2 contents are being constructed. The apparatus is mobile. Field trials have shown good agreement between the sodium concn. found in simultaneous photometric and electrical pptn. tests; 7% of the sodium salts in the pulverised fuel was carried as aerosol, and 30% of this amount was deposited in the superheater.

A. R. PEARSON

2866. **Use of fluoroboric acid in quantitative analysis. Determination of potassium and sodium in mixtures of chlorides.** I. G. Rŷss and É. L. Hilus (Dniepropetrovsk Inst. of Railway Transport Engineers). *Zavod. Lab.*, 1958, **24** (11), 1349–1352.—The rate of dissolution of KBF_4 in 10% NH_4BF_4 soln. is so low and that of $NaBF_4$ is so high that treatment for 5 min. with 2·5 ml of this soln. can be used for the quant. separation of Na and K in the mixture obtained by evaporation of a soln. of the Cl^- with 40% HBF_4 soln.

G. S. SMITH

2867. **Amperometric titration of potassium with sodium tetraphenylboron.** W. R. Amos and R. F. Sympson (Dept. of Chem., Univ., Athens, Ohio, U.S.A.). *Anal. Chem.*, 1959, **31** (1), 133–135.—Potassium in concn. from $2{\cdot}2 \times 10^{-3}$ to 2×10^{-2} *M* in acetate buffer (0·8 *M*) can be titrated directly with 0·05 to 0·15 *M* Na tetraphenylboron by making 17 to 25 measurements of the anodic depolarisation current (*I*) of the titrant at the dropping-mercury electrode. The end-point (sharp increase in *I* caused by free tetraphenylboron) is located by extrapolation of the two linear curves [*I vs.* vol. (ml) of titrant] to their intersection. The error is within $\pm$ 1·0% and a complete titration takes $\simeq$ 2 to 3 hr. Chloride interferes at concn. $>$ 0·32 *M*.

W. J. BAKER

2868. **Spectrographic determination of potassium in coal ash.** A. F. Rekus (Baltimore Gas & Electric Co., Baltimore, Md., U.S.A.). *Appl. Spectroscopy*, 1958, **12** (5), 141–142.—Indium is a useful internal standard for determining K in coal ash by a d.c. arc spectrographic procedure, and may also be useful for determining Na. Cyanogen bands occur in the region of the analysis and internal standard lines and are suppressed by using a Li_2CO_3 matrix. The coeff. of variation was 6% for 23 samples with a K_2O content ranging from 0·8% to 2·3%. The method may also be applicable to fly ash, slag and boiler deposits.

K. A. PROCTOR

2869. **Separation of radioactive caesium in biological materials.** Nobonu Yamagata and Toshiko Yamagata (Kirui Coll. of Technol., Gunma Univ., Kiriu). *Bull. Chem. Soc. Japan*, 1958, **31** (9), 1063–1068 (in English).—A simple and rapid separation of ^{137}Cs in biological materials is carried out in three stages—(*i*) phosphate elimination, (*ii*) ammonium molybdophosphate pptn., and (*iii*) chloroplatinate pptn. The over-all yield of Cs is $>$ 85%, and the decontamination from other fission activities is $> 10^5$ for ^{144}Ce and ^{89}Sr. The interference of ^{87}Rb and ^{40}K is negligible in the determination of ^{137}Cs in biological materials by β-counting. The procedures for urine, cereals, vegetables, dried milk and soil are described.

I. JONES

2870. **Colorimetric analysis with organic reagents. V. Spectrophotometric determination of copper with quinoline-2-carboxyaldoxime.** Nobuichi Oi (Fac. of Engng, Toyama Univ., Takaoka). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1958, **79** (11), 1327–1330.—Quinoline-2-carboxyaldoxime (**I**) reacts with Cu^I (orange coloration), Cu^{II} (green), Pd (yellow) and Hg^{II} (white ppt.) in weakly acid soln. The max. absorption of the Cu^+ - **I** complex lies at 467 mμ, where the absorption of **I** is negligibly small, and remains unchanged in the pH range 4·5 to 8·5. The extinction is proportional to the amount of Cu for $<$ 10 μg per ml. Reduction of Cu^{2+} is effected with hydroxylamine hydrochloride (**II**). The sample soln. is mixed with **II** soln. (10%, 1 ml), acetate buffer of pH 5·2 (1 *M*, 25 ml) and **I** in ethanol (0·1%, 10 ml), and made up to 50 ml and the extinction measured after 10 min. Interference is caused by Pd, coloured cations (*e.g.*, $>$ 5 mg of Ni, $>$ 2 mg of Cr^{3+}, $>$ 5 mg of Fe^{3+} and V^{5+}), CN^-, SCN^- and NO_2^-.

VI. Spectrophotometric determination of copper with quinoline-2-carboxyaldoxime by the extraction method. Nobuichi Oi. *Ibid.*, 1958, **79** (11), 1331–1333.—Univalent Cu is extracted with higher alcohols in the presence of **I** at pH 5. The max. absorption lies at 478 mμ and the extinction is proportional to the amount of Cu for $<$ 10 μg per ml. This method is applicable to the determination of Cu in iron and steel. The error is $\simeq$ 1·5% for $>$ 0·002% of Cu. *Procedure for iron and steel*—Dissolve the sample (0·1 g) in H_2SO_4 - HNO_3 - water (2:1:10, by vol.) (10 ml). Add ammonium citrate soln. (20%, 2 ml), **II** soln. (10%, 5 ml), aq NH_3 (1:1) and water to produce $\simeq$ 30 ml of a soln. of pH 4·5 to 5·0. Heat at 80° for 10 min., cool, add **I** soln. (0·1% in ethanol) (2 ml), dilute to 50 ml and shake with *iso*amyl alcohol (10 ml). After 10 min., centrifuge the organic layer and measure the extinction. Prepare the working curve with electrolytic iron and standard soln. of Cu.

K. SAITO

2871. **Photometric determination of copper [in light alloys] with violuric acid.** M. Ziegler (Anorg.-chem. Inst., Univ. Göttingen, Germany). *Z. anal. Chem.*, 1958, **164** (4), 387–390.—From 0·02 to 10% of Cu in the alloy can be determined spectrophotometrically as the violurate at 415 mμ and pH 4·5 to 5·5. *Procedure*—Dissolve the sample in dil. HCl, warm the soln. with conc. HNO_3 (5 to 10 drops) and dilute to 250 ml. To an aliquot (50 ml) add 0·5% aq. violuric acid soln. (20 ml) followed by 15% aq. Na_2CO_3 soln., with stirring, until a pale-yellow coloration appears. After further addition of acetate buffer (10 ml) and 10% aq. KF soln. (2 ml), place the soln. under vacuum for $\simeq$ 30 sec. to remove CO_2 and then measure the extinction, within 4 hr., in a 1- or 5-cm cell. Amounts of Ni $\not>$ 4% do not interfere; the max. permissible concn. of other elements are listed. Beer's law is not strictly followed, but the curve is sufficiently accurate for routine analyses.

W. J. BAKER

2872. **Absorptiometric determination of traces of copper in rubber chemicals and products.** R. C. Madan (Firestone Tyre & Rubber Co., Ltd., Brentford, Mddx., England). *Rubb. Age*, 1958, **84**, 451–

454.—The sample is treated with Zn dibenzyldithiocarbamate and the complex formed is simultaneously extracted with CCl_4 in an acid medium. The colour is measured spectrophotometrically; Bi, Ni, Co, Hg, Ag and Fe^{3+} interfere.

S.C.I. Abstr.

2873. **Micro-detection with the aid of ion-exchange resin beads. Detection of silver by catalytic reduction of ter- and quadri-valent manganese.** Takeo Murase (Ind. Res. Inst. Aichi Prefecture, Chikusa-ku, Nagoya). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1958, **79** (11), 1389–1393.—The detection of Ag by Lang's catalytic reduction is improved by the use of anion-exchange resin beads containing Mn^{III} and Mn^{IV}. Amberlite IRA-411 (Cl) (30 to 50 mesh, wet vol. $\simeq$ 0·5 ml) is treated with $KMnO_4$ soln. (0·05%, 10 ml) for 30 min., washed with water, treated with $MnSO_4$ (0·2 *M*, a few ml) for 30 min. and washed. One drop each of the sample and 5 *N* HCl are mixed on a porcelain plate and one bead of the resin is added. In the presence of $>$ 0·006 μg of Ag ($>$ 0·25 μg per ml), the colour changes to pale yellow within 100 min.

K. Saito

2874. **Solvent extraction of beryllium as acetylacetonate.** Masayuki Tabushi (Shigematsu Lab., Kyoto Univ., Kyoto, Japan). *Bull. Inst. Chem. Res. Kyoto Univ.*, 1958, **36** (6), 156–162 (in English).—The determination of Be by acetylacetone (**I**) and Eriochrome cyanine R (**II**) has been studied, and suitable procedures are given. Beryllium acetylacetonate is extracted quant. by $CHCl_3$ from aq. soln. at pH 6 to 9. The absorbance of the $CHCl_3$ soln. can be measured at 295 mμ, after removing excess of **I** by shaking with dil. NaOH soln. Alternatively, the $CHCl_3$ soln. can be evaporated, the residue decomposed with $HClO_4$ and Be determined with **II**. *Method* (*i*)—To the test soln. containing not more than 5 μg of Be add 2 ml of 10% EDTA (disodium salt) soln., 2·5 g of NaCl, and 1 ml of 5% acetylacetone soln. Adjust the pH to 7 to 8, dilute to 50 ml and extract with $CHCl_3$ (10 + 5 + 5 ml). Wash the extract with two 50-ml portions of 0·1 *N* NaOH, dehydrate with a little anhyd. Na_2SO_4 and measure the absorbance at 295 mμ. The recovery is not greater than 97%, but if no Mn^{II}, Cr^{III} or Ti^{IV} is present the EDTA and NaCl can be omitted, when the recovery is 100%. *Method* (*ii*)—Evaporate the $CHCl_3$ soln. obtained as above in a small beaker and fume to dryness with $HClO_4$ to remove excess of **I**. Add 5 ml of 5% Na acetate soln., 1 ml of 10% EDTA (disodium salt) soln. and 5 ml of 0·1% **II**. Adjust the pH to 9·7 to 9·8 with *N* NaOH, transfer to a 50-ml flask, dilute to the mark and measure the extinction at 510 mμ.

H. M.

2875. **Polarography of beryllium.** G. Venkataratnam and B. S. V. Raghava Rao (Chem. Dept., Andhra Univ., Waltair, India). *J. Sci. Ind. Res., India, B*, 1958, **17** (9), 360–362.—In 0·5 *M* LiCl soln. beryllium gives two well-defined polarographic waves. The half-wave potentials of these waves are dependent on pH ($-$ 1·79 V and $-$ 2·00 V *vs.* the S.C.E. at pH 2·38, and $-$ 1·70 V and $-$ 1·91 V at pH 4·14). The diffusion current for the first reduction is proportional to concn. of Be^{2+} up to 5 $\times$ 10^{-3} *M*.

T. R. Andrew

2876. **Spectrophotometric determination of beryllium and fluoride using Chrome azurol S.** L. Silverman and M. E. Shideler (Atomics Internat., North American Aviation, Inc., Canoga Park, Calif., U.S.A.). *Anal. Chem.*, 1959, **31** (1), 152–155.—From 1 to 30 μg of F^- or 0·2 to 10 μg of Be in 50 ml of soln. can be determined spectrophotometrically to within $\pm$ 1 μg or $\pm$ 0·2 μg, respectively. The purplish-blue complex of Be with Chrome azurol S is formed at pH 6 in the presence of a pyridine - HCl buffer to increase the sensitivity of the dye to Be and of the metal - dye complex to F^-. The extinction of the soln. is measured at 575 mμ (5-cm cell) against a reagent blank free from Be and F^-. In the presence of F^-, the decrease in extinction is proportional to the concn. of F^-. The standard graphs for F^- and Be are linear, and the coloured complex is stable for 5 hr. All interfering ions (especially U^{6+} and Fe^{3+}) should be removed initially, preferably by distillation of the fluoride or by recovery of Be by an ion exchange - evaporation procedure. (*Cf.* Wood, *Anal. Abstr.*, 1955, **2**, 1769.)

W. J. Baker

2877. **Spectrophotometric determination of magnesium in aluminium alloys.** G. Selzer and M. Ariel (Fac. of Sci., Dept. of Chem., Israel Inst. of Technol., Haifa). *Anal. Chim. Acta*, 1958, **19** (5), 496–502.—Magnesium in aluminium alloys is determined spectrophotometrically with Eriochrome black T, after separation by pptn. with NaOH and further removal of interfering ions with thioacetamide (**I**). A controlled amount of KCN is added to remove interference by traces of Cu^{2+} left after treatment with **I**. The extinction of a blank is measured against H_2O at 520 mμ, and a second wavelength, in the region 690 to 720 mμ, is found at which the extinction is the same. The extinctions of the test soln. are measured against the blank at the two wavelengths, and the difference between these readings is referred to a standard curve, to give the amount of Mg in the test soln., which is converted to percentage by multiplying by a dilution factor.

T. R. Andrew

2878. **Mass-spectrometric isotopic analysis of magnesium, zinc and cadmium using the reduction of oxides by carbon in the ion-source furnace.** D. D. Gvozdanović. *Bull. Inst. Nucl. Sci. "Boris Kidrich", Belgrade*, 1958, **8** (148), 151–155 (in English).—Magnesium oxide powder was mixed with an equal amount of carbon powder obtained by the decomposition of pure organic solvents. The current was gradually raised from 0·5 to 20 mA so that the furnace reached a temp. of 1500° in the hottest region. The reduction lasted 20 min. with a pumping rate of 20 litres per sec. in the source region. When the ionising current was 1 mA the electron energy was 40 eV and this was increased to 2000 eV. Examples of mass spectra for Mg, Zn and Cd, obtained by electrostatic or electromagnetic scanning and recorded on a cathode-ray tube, are given. A stable and intense ion current was obtained and the manipulation of the sample was simplified. Analyses could be carried out even with fractions of a milligram, and the results were well within experimental error (1%) for the mass spectrometer. It is suggested that the method can be used for the analysis of all elements that can be reduced in this way and that are volatile at a temperature of $<$ 1600°.

K. R. C.

2879. **New specific test for calcium.** D. Goldstein and C. Stark-Mayer (Lab. da Prod. Mineral, Min. Agric., Rio de Janeiro, Brazil). *Anal. Chim. Acta*, 1958, **19** (5), 437–439.—Glyoxal bis-(*o*-hydroxyanil) reacts with Ca in alkaline soln. to give a red ppt.

Interference by Ba or Sr is eliminated by addition of Na_2CO_3 soln. after formation of the ppt. Of 33 cations studied, only Co^{2+} and Ni^{2+} give a similar red ppt. The Ca complex is sol. in $CHCl_3$ while those of Co and Ni are not. As little as 0·05 μg of Ca may be detected in 0·05 ml. The prep. of the reagent from *o*-aminophenol and glyoxal is described. T. R. ANDREW

2880. **Volumetric determination of calcium and magnesium with ethylenediaminetetra-acetic acid in the presence of phosphate.** C. Cimerman, A. Alon and J. Mashall (Israel Inst. of Technol., Dept. of Chem., Haifa, Israel). *Anal. Chim. Acta*, 1958, **19** (5), 461–466.—By titrating with a mixed soln. of EDTA (disodium salt) and EDTA (monozinc salt), Mg and Ca may be determined in the presence of moderate amounts of phosphate; 2 to 10 mg of Ca in the presence of $>$ 60 mg of P_2O_5; up to 2 mg of Mg in the presence of $>$ 20 mg of P_2O_5; and up to 5 mg of Mg in the presence of $>$ 10 mg of P_2O_5. To prepare the reagent, dissolve 4·069 g of ZnO in HCl (4 ml) and dilute to 200 ml. Add 22·332 g EDTA (disodium salt) and aq. NH_3 until the soln. becomes clear (pH 8 to 9); dilute to 1 litre and standardise against 0·01 *M* $CaCl_2$ at pH 10. T. R. ANDREW

2881. **Rapid quantitative determination of calcium, magnesium and sulphate in common salt by means of chelatometric titrations.** B. Riva (Inst. Appl. Chem., Univ. Cagliari, Italy). *Ann. Chim., Roma*, 1958, **48** (11), 950–959.—To determine Ca^{2+}, add *N* NaOH (20 ml) and murexide (**I**) (0·5 g) to a 200-ml aliquot containing 1 to 5 g of NaCl, and titrate with 0·02 *M* EDTA. To determine Mg^{2+}, add conc. HCl (1 to 2 ml) to this soln. after titration, warm (40° to 50°) to decompose **I**, adjust to pH $<$ 10 with $\simeq$ 5 ml of buffer soln. [54 g of NH_4Cl and 350 ml of aq. NH_3 (*d* 0·98), diluted to 1 litre] and titrate with 0·02 *M* EDTA, with Eriochrome black T (**II**) as indicator. Alternatively, determine Ca^{2+} as before, then warm a second aliquot to 40° to 50°, add buffer soln. (5 ml) and 0·02 *M* EDTA in vol. corresponding to that required to complex the Ca^{2+}, and titrate further with 0·02 *M* EDTA, with **II** as indicator. To determine SO_4^{2-}, add to an aliquot at the b.p. a vol. of 0·02 *M* $BaCl_2$ corresponding to the total vol. of 0·02 *M* EDTA used in titrating the Ca^{2+} and Mg^{2+}, then add 5 ml in excess, boil for a few minutes, leave for 1 hr., warm to 40° to 50°, adjust to pH $<$ 10, add a vol. of 0·02 *M* EDTA equal to the total used in determining Ca^{2+} and Mg^{2+} and titrate the excess of Ba^{2+} with 0·02 *M* EDTA, with **II** as indicator. The method is rapid and results compare favourably with those determined by conventional means. A. G. COOPER

2882. **Rapid separation of radioactive strontium by solvent extraction with TTA [2-thenoyltrifluoroacetone] - hexone [*iso*butyl methyl ketone].** Toshiyasu Kiba and Shigeru Mizukami (Chem. Dept., Univ., Kanazawa). *Bull. Chem. Soc. Japan*, 1958, **31** (9), 1007–1013 (in English).—A two-step process for the rapid separation of radioactive Sr in mixed fission products is described. A soln. of the material is adjusted to pH 7 and extracted with 0·05 *M* 2-thenoyltrifluoroacetone in benzene, when rare-earth elements and Y enter the organic phase. The aq. phase is adjusted to pH 8 and extracted with 0·05 *M* 2-thenoyltrifluoroacetone in hexone when radioactive Sr passes completely into the organic phase. Less than 5% of the Ru present in the soln. is extracted at the same time. I. JONES

2883. **Separation of fission products by liquid - liquid extraction with cupferron - chloroform.** Toshiyasu Kiba and Mitsue Kanetani (Chem. Dept., Kanazawa Univ.). *Bull. Chem. Soc. Japan*, 1958, **31** (9), 1013–1016 (in English).—When a soln. (*N* in HCl) containing fission products together with other non-radioactive metal ions is adjusted to pH 4 and extracted with cupferron - $CHCl_3$, radioactive Sr and Cs remain in the aq. phase. Radioactive Sr then passes into the organic phase on extraction with 2-thenoyltrifluoroacetone (0·05 *M*) in *iso*butyl methyl ketone at pH 8. I. JONES

2884. **Separation of strontium-90 from calcium.** P. S. Davis (Austr. A.E.C., Res. Estab., Lucas Heights, New South Wales). *Nature*, 1959, **183**, 674.—The adsorption of Ca and Sr on "Zeo-Karb 225" was studied over the pH range 3 to 7. At pH 5·25 all the Sr of a mixture of 25 mg of Sr, 25 mg of Ca and 1 g of EDTA (disodium salt) is retained on a 10-ml column. The recovery was checked by labelling with ^{45}Ca and elution of the Sr with 3 *N* HCl. The contamination was $< 1\%$. S. BAAR

2885. **Strontium-90 by an ion-exchange method.** E. A. Bryant, J. E. Sattizahn and B. Warren (Univ. Calif., Los Alamos Sci. Lab., N.M.). *Anal. Chem.*, 1959, **31** (3), 334–337.—The separation of ^{90}Y, formed by decay of ^{90}Sr in fission-product samples at least 10 days old, from Sr and accompanying fission products ^{140}Ba and ^{140}La is described. Large amounts of Fe and U are removed by adsorption from HCl on to an anionic resin, and ^{95}Zr, ^{103}Ru and ^{131}I by $HClO_4$ digestion and pptn. with aq. NH_3. The filtrate is passed through a cationic resin (NH_4^+), which is then washed with $HClO_4$; Y, La and other rare-earth metals are eluted with ammonium α-hydroxy*iso*butyrate (**I**) soln. at pH 6. After washing the resin with H_2O, the start of the ^{90}Y growth period is recorded and the column is sealed off for 2 days; ^{90}Y is then removed with **I** at pH 3·55 and collected on a second column containing La carrier in HCl soln. Complete elution marks the end of the growth period and the beginning of the decay period for ^{90}Y. The second column is washed with H_2O, and 0·5 *M* NH_4ClO_4 is passed through until the percolate is at pH 5. After washing the column with H_2O, **I** at pH 3·55 is passed through and the eluate is collected for β-counting on a heated counting plate. The radiochemical yield is $> 97\%$. P. D. PARR-RICHARD

2886. **Application of EDTA to the determination of zinc—accuracy and precision of analysis.** J. Lacy (African Explosives & Chem. Industries, Ltd., P.O. Northrand, Transvaal). *J. S. Afr. Chem. Inst.*, 1958, **11** (2), 42–44.—An aliquot of the dissolved sample ($\simeq$ 0·15 g of Zn) is boiled with 5 ml of conc. HNO_3 in a 500-ml flask. Conc. aq. NH_3 is added to the slightly cooled soln. until a permanent ppt. forms, and 10 ml in excess. After cooling to 60° and addition of 4 ml of 20% KCN soln., the soln. is filtered and the ppt. is washed with 2% aq. NH_3. The filtrate and washings are diluted to about 300 ml with H_2O and 25 ml of 4% formaldehyde soln. is added, followed immediately

by Eriochrome black T soln. (1 to 1·5 ml). The soln. is titrated, as rapidly as possible if Cu is present, with 0·1 *M* EDTA until the colour changes from wine red to blue. Determinations of Zn by this method can be completed in 25 min., and are more accurate and precise than those by the ferrocyanide method. A. M. SPRATT

2887. **Iodimetric determination of zinc in ammonium hydrogen difluoride buffer medium. Rapid estimation of total zinc oxide in mixed paint pigments.** K. C. Seal (Gov. Test House, Alipore). *Indian J. Appl. Chem.*, 1958, **21**, 125–128.—The use of an $NH_4F.HF$ buffer for the iodimetric determination of Zn ensures a suitable pH and prevents interference from Fe^{3+}. An amount of $K_3Fe(CN)_6$ (0·1 *M*) is added such that the excess remaining after the reaction is < 2 millimoles per litre. Under these conditions, the stoicheiometric amount of Zn is multiplied by an empirical factor of 1·020. The titration is rapid and the end-point sharp and stable. The method is useful for the determination of total ZnO in mixed paint pigments. Moderate concn. of Fe^{3+}, Pb^{2+} and Cr^{3+} do not interfere. S.C.I. ABSTR.

2888. **Post-precipitation of zinc with homogeneously precipitated copper sulphide.** G. K. Schweitzer and G. M. Correll (Dept. of Chem., Univ. of Tennessee, Knoxville, U.S.A.). *Anal. Chim. Acta*, 1958, **19** (5), 467–470.—The co-pptn. of ZnS on CuS pptd. with thioacetamide has been studied as a function of time, temp., and acidity. The amount of Zn found in the ppt. increases with increasing Zn concentration, digestion time and temp., and decreasing acidity, rising to about 25% of the total Zn present on digesting for 45 min. at 98° in 0·1 *N* acid. By working at acidities above 0·5 *N* and temp. ≯ 80°, post-pptn. may be reduced to negligible proportions. T. R. ANDREW

2889. **Determination of cadmium by flame photometry.** P. T. Gilbert, jun. (North American Aviation, Inc., Downey, Calif., U.S.A.). *Anal. Chem.*, 1959, **31** (1), 110–114.—From 50 to 10000 p.p.m. of Cd can be determined to within ≃ 0·1% by excitation in the air - hydrogen flame and measurement at 326·1 mμ (slit width 0·15 mm) in a Beckman flame photometer with photomultiplier attachment. Background and/or radiation interference by Al, Fe, Cu and Ca can be eliminated or minimised by using the method of successive dilutions. The working curve for Cd is approx. linear below 500 p.p.m. The lower limit of determination is 0·5 p.p.m., the error at these very low concn. being ≃ 0·1 p.p.m. In comparison with other flames, the air - hydrogen flame ensures lowest background emission and max. intensity for Cd at 326·1 mμ, this line showing negligible self-absorption compared with the resonance line at 228·8 mμ. W. J. BAKER

2890. **Use of "cadion" [*p*-nitrophenyldiazoaminoazobenzene] in analytical chemistry. II. Spectrophotometric micro-determination of mercury.** P. Chavanne and C. Geronimi. *Anal. Chim. Acta*, 1958, **19** (5), 442–447 (in French).—In 50% v/v ethanol soln. (*N* in KOH), cadion reacts with Hg^{2+} to give a coloured soln. with max. absorption at 460 mμ. The sensitivity of the procedure is increased by measuring the decrease in absorption of the free reagent at 560 mμ. Of 34 ions studied, only AsO_3^-, CN^-, Sn^{2+} and Sb^{3+} interfere at concn. 10 times that of the Hg^{2+}. At 560 mμ, 1·5 p.p.m. of Hg^{2+} gives rise to a difference in extinction of 1·0 in a 1-cm cell. Beer's law is followed up to 1·4 p.p.m. of Hg, and under the conditions specified the mean square error is 1·2%. T. R. ANDREW

2891. **Spectrophotometric determination of boron with morin.** Akira Murata and Fumio Yamauchi (Fac. of Engng, Shizuoka Univ., Oiwake, Hamamatsu). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1958, **79** (11), 1454–1458.—When a neutral soln. containing BO_3^{3-} (< 10 μg of B) is evaporated to dryness with 3 *N* HCl (1 ml), 4% oxalic acid (1·5 ml) and 0·1% morin in ethanol (1 ml), the yellow residue is extractable with acetone (3 ml). The extinction of the acetone soln. at 425 mμ is proportional to the amount of B (0·5 to 10 μg). Since inorganic salts are insol. in acetone, the distillate obtained by Chopin's methyl borate distillation is used for this procedure after neutralisation. The development of the colour is complete after heating the acetone extract at 55° ± 2° for 1 hr. The residue is again extracted with acetone (25 ml) for photometry. The colour gradually fades at a constant rate both in the light and in the dark at a given temp. K. SAITO

2892. **Quantitative determination of boron in minerals by the neutron - gamma method.** Yu. V. Galuzo. *Izv. Vyssh. Ucheb. Zaved., Neft' i Gaz*, 1958, (1), 41–44; *Ref. Zhur., Khim.*, 1958, (23), Abstr. No. 77,292.—The large neutron capture cross-section (σ_s = 753 barns) and low γ-radiation energy of B cause the curve of depth of insertion (in the bore) of the neutron probe *vs.* induced γ-activity (number of counts per min.) to reveal a definite minimum of γ-activity at the abscissa corresponding to the passage of the probe past the B-containing layer. A test experiment has shown that the sensitivity of the method is very high and corresponds to a content of 0·2 to 0·3% of B in the mineral. C. D. KOPKIN

2893. **Determination of boron in glass by neutron absorption using a radium - beryllium source.** Yuzuru Kusaka (Dept. of Chem., Kōnan Univ., Higashinada-ku, Kōbe). *Bull. Chem. Soc. Japan*, 1958, **31** (8), 917–920 (in English).—The source is a mixture of radium bromide and Be, sealed in a platinum tube which is embedded in a block of paraffin wax. The radioactivity of a silver plate after a timed exposure was measured with a sample of borosilicate-glass powder (diluted with Al_2O_3) interposed between it and the source. Results were referred to a calibration curve. The error was ± 10% at 2% of B falling to ± 2% for 24% of B. S.C.I. ABSTR.

2894. **Amperometric determination of boric acid.** V. I. Bogovina and V. G. Selivanov (Dnepropetrovskiĭ Khim.-Tekhnol. Inst. im. F. É. Dzerzhinskogo). *Zavod. Lab.*, 1958, **24** (10), 1200–1202.—Platinum wire serves as the anode and a saturated calomel electrode as the external cathode. The titration is carried out with NaOH soln. at 0·55 V and takes only 4 to 5 min. *Procedure*—Mix ground elementary boron (< 100 mesh) (0·1 g) with 2·0 g of NaOH pellets and fuse in a covered iron or silver crucible. Dissolve the cooled melt in 50 ml of HCl (1 : 1), wash the crucible well with water, and make up the washings and main soln. to 200 ml with water. Add 20 ml of NaOH soln. (1 : 2) and, while stirring, 10 g of $BaCO_3$ powder so that a slight excess is present. Boil the mixture under reflux for 5 min. until all the iron hydroxides

are pptd. Allow the ppt. to settle for 30 min. before filtering. Acidify the filtrate ($>$ 400 ml) to litmus paper with HCl (1:3); boil under reflux for 10 min., cool quickly, and make up the soln. to 500 ml. Transfer a 50-ml aliquot ($\equiv$ 0·010 g of B) to a titration vessel, and add 6 or 7 drops of indicator mixture (0·05 g of methyl red, 0·1 g of bromocresol green, 0·30 g of phenolphthalein and 0·30 g of thymolphthalein in 100 ml of ethanol). Apply an e.m.f. of 0·55 V, and when the microammeter needle sets to zero position add 0·1 *N* NaOH dropwise, stirring with a rotating platinised electrode, until the soln. becomes green, indicating neutralisation of free HCl. Add 10 ml of invert sugar soln. or 1 g of mannitol, and when the microammeter needle has returned to its zero position, finish the titration with 0·1 *N* NaOH. Mineral acids such as HCl, H_2SO_4 and HNO_3 do not interfere if they are first neutralised. Weak acids, such as carbonic acid, must be removed. This method can be applied to the determination of boric acid in nickel electrolytes after separating nickel as the dimethylglyoxime complex. W. Roubo

2895. **Titrimetric determination of aluminium with ethylenediaminetetra-acetic acid in the presence of iron, copper, titanium, manganese, calcium, magnesium and phosphate.** C. Cimerman, A. Alon and J. Mashall (Israel Inst. of Technol., Dept. of Chem., Haifa). *Talanta*, 1958, **1** (4), 314–328.—The rapid method described is based on the treatment of the weakly acid soln. (5 to 15 mg of Al) with enough EDTA to chelate the Al, Cu and Fe. To avoid excess, which would also chelate Mn, Ca, Ti and Mg, a specified spot-test is applied. The soln. is boiled to stabilise the Al chelate, then cooled and made faintly alkaline with aq. NH_3 and treated with $ZnSO_4$ soln. and HCl until orange to methyl orange. The soln. is passed through a column of Amberlite IR-120 (ammonium form); the Al, Cu and Fe chelates pass through, while the unchelated Mn, Ca, Ti and Mg are retained on the resin. The eluate and washings are treated with *iso*propyl alcohol, ammonium acetate and triethanolamine, and the excess of EDTA is titrated with $ZnSO_4$ soln. at $< 10°$, with Eriochrome black T as indicator. Sodium fluoride (1·5 g) is then added, and the mixture kept near the boiling-point for 20 min. This liberates the EDTA from the Al chelate, the liberated EDTA being titrated at $< 10°$ with $ZnSO_4$ soln. Full experimental details are given. Reducing agents interfere if Cu or Fe is present, and must be oxidised with bromine water. Oxalates, tartrates, citrates, pyrophosphates and fluorides interfere, and must be removed by evaporation with H_2SO_4. Chloride, sulphate, nitrate and acetate ions do not interfere. The following max. amounts (mg) can be present without interference—Fe, 8; Cu, 3; Ti, 1; Mn, Ca or Mg, 20; phosphate as P_2O_5, 40. A single determination occupies 90 to 105 min., but much less time is required if the determinations are carried out in series. Quoted results show relative errors of from $-$ 0·5% to $+$ 0·9%.
R. E. Essery

2896. **Flame-spectrometric determination of aluminium in aluminium silicates.** S. Schmidt and K. Konopicky. *Ber. dtsch. keram. Ges.*, 1958, **35**, 317–321.—Sulphuric acid and Na_2O had a marked and irregular effect on the emission of the 484-mμ line of Al and the test soln. must therefore be prepared from the pptd. hydroxides. The interference by Fe_2O_3 and TiO_2 was sufficient to necessitate their separate determination so that their concn. can be reproduced in the calibrating soln.; HCl and NH_4Cl in amounts, respectively, of $\leq$ 10 ml of 18% HCl soln. per 100 ml and $\leq$ 0·5 g per 100 ml had constant effects; CaO, MgO and K_2O had little effect on the emission. *Procedure*—The sample (0·5 g) is decomposed by evaporating to dryness with HF plus H_2SO_4, the residue is dissolved in conc. HCl, and the hydroxides pptd. with aq. NH_3. The ppt. is dissolved in 50 ml of 18% HCl and the soln. is made up to 250 ml. An aliquot is used for the colorimetric determination of Fe_2O_3 and TiO_2. To 50 ml of this soln. are added 4 ml of *n*-butanol (flame activator) and 0·5 g of NH_4Cl, and the Al_2O_3 is determined by direct flame-photometry against a series of standard soln. containing 0 to 40 mg of Al_2O_3 per 100 ml and from combinations of quantities of Fe_2O_3 and TiO_2 varying from 1·5 to 3 and from 1 to 2·75 mg per 100 ml, respectively. For a content of 35% of Al_2O_3, an accuracy within $\pm$ 0·5% is claimed.
J. A. Sugden

2897. **Systematic analysis of the impurities in aluminium and iron (obtained by zone-melting) by irradiation in the atomic pile.** J. Gaittet and P. Albert. *Compt. Rend.*, 1958, **247** (21), 1861–1863.—A scheme of analysis for the separation and determination of $\simeq$ 30 elements in high-purity aluminium or iron is outlined. The sample (1 g) is irradiated for one week (flux of 10^{12} neutrons per sq. cm per sec.), cleaned with dil. acid or alkali, and dissolved in HCl (for aluminium) or HNO_3 (for iron). Iron is complexed with oxalic acid before the pptn. of sulphides with H_2S in 5 *N* HCl. The effectiveness of the separation and the accuracy of the results depend on the controlled addition (time and concn.) of non-radioactive carriers for each element (*e.g.*, Cu and As are added before the pptn. of the acid sulphides, Ca and Mg at the time of pptn. of the carbonates). Each separated radio-isotope is identified and determined by means of its decay period and type and energy of radiation; γ-ray spectrometry is used for long-lived radio-isotopes. The lower limit of sensitivity is $\simeq$ 1 in 10^7.
W. J. Baker

2898. **Determination of indium by flame photometry.** P. T. Gilbert, jun. (N. Amer. Aviation Inc., Downey, Calif., U.S.A.). *Spectrochim. Acta*, 1958, **12** (4), 397–400.—Observation of the spectrum of In in air - hydrogen and oxy-hydrogen flames, with a Beckman DU spectrophotometer fitted with Model 9200 flame attachment and 1P28 photomultiplier, is described. The bands at 4511, 4102 and 3256 A could all be used for quant. work at concn. $>$ 0·01 p.p.m. The 4511-A emission with the oxy-hydrogen flame was strictly proportional to concn. up to 100 p.p.m. and at this wavelength In in soln. was detectable down to the 0·01 p.p.m. level. Sensitivity decreases in the presence of HCl.
E. G. Cummins

2899. **Radiochemical separations of indium.** D. N. Sunderman, I. B. Ackermann and W. W. Meinke (Univ., Ann Arbor, Mich., U.S.A.). *Anal. Chem.*, 1959, **31** (1), 40–44.—The radiochemical separations of In by (*i*) sulphide pptn. at pH 1 to 3, (*ii*) extraction of the bromide with diethyl ether, (*iii*) extraction with 0·5 *M* 2-thenoyltrifluoroacetone at pH 2·5, and (*iv*) anion exchange in 6 *M* HCl, have been evaluated for efficiency of separation from $\simeq$ 15 different tracers. None of the procedures is specific, each giving decontamination factors from

10 to 10^4 for most elements. With optimum conditions, a 1-hr. anion-exchange procedure gives decontamination factors of $\simeq 10^3$. A 15-min. bromide extraction (yield of In 90 to 95%) can be followed by method (*iii*) (yield of In 70 to 80%) or by anion-exchange separation, according to the composition of the sample. Method (*i*) should be used mainly for the final preparation of counting-plates or as the initial step in determining carrier-yield by dissolution of sulphide and pptn. of indium hydroxide. W. J. BAKER

2900. **Ultra-micro method for the estimation of the rare earths by complexometric titration.** E. A. C. Crouch and I. G. Swainbank (A.E.R.E., Harwell, Berks., England). A.E.R.E. Report C/R **2843**, 1959, 6 pp.—The method of Körbl and Přibil (*cf. Anal. Abstr.*, 1957, **4**, 2072) for use with milligram amounts has been adapted for microgram quantities. Nitric acid soln. of the rare-earth metals are buffered and titrated with EDTA (disodium salt) soln. to a photometrically detected end-point. Results are accurate to within ± 2%. G. S. ROBERTS

2901. **Quantitative spectrographic analysis of the rare-earth elements. IX. Determination of rare-earth impurities commonly associated with purified samarium, gadolinium, terbium and yttrium.** R. N. Kniseley, V. A. Fassel, B. B. Quinney, C. Tremmel, W. A. Gordon and W. J. Hayles (Atomic Res. Inst., Iowa State Coll., Ames, U.S.A.). *Spectrochim. Acta*, 1958, **12** (4), 332–337.—Further work with the Jarrell-Ash 3·4-metre Wadsworth grating spectrograph (*cf.* Fassel and Wilhelm, *Brit. Abstr. C*, 1949, 7) on the determination of impurities (< 1%) in purified samarium, gadolinium, terbium and yttrium is described. The need for careful selection of appropriate line-pairs is stressed. Tabulated data give the detection limits and determination ranges for Ho, Eu, Nd, Dy, Y, Sm and Tb in gadolinium, Nd and Gd in samarium, Dy, Y, Sm and Gd in terbium, and Sm, Gd, Tb, Dy, Ho and Er in yttrium, regions of interference being noted.

X. Determination of rare-earth impurities commonly associated with purified thulium, ytterbium, lutetium and scandium. R. N. Kniseley, V. A. Fassel, R. W. Tabeling, B. G. Hurd and B. B. Quinney. *Ibid.*, 1959, **13** (4), 300–303.—Data are given for suitable line-pairs in the emission-spectrographic determination of rare-earth impurities in purified rare-earth matrices, *e.g.*, Y, Ho, Er, Yb and Lu in thulium; Sm, Y, Er, Tm, Lu and Sc in ytterbium; Tm, Yb, Sc and Y in lutetium; Y, Tm, Yb and Lu in scandium. The basic procedure uses d.c. carbon arc excitation of rare-earth oxide - graphite mixtures and the accuracy is claimed to be within ± 5%.

E. G. CUMMINS
H. M.

2902. **Gas-chromatographic investigations. [Solubility of carbon dioxide in adsorbed acetone.]** G. Székely, T. Kormány, G. Rácz and G. Traply (Lehrstuhl f. Phys. Chem., Tech. Univ., Budapest). *Period. Polytech.*, 1958, **2** (4), 269–274 (in German).—The apparent solubility of CO_2 in acetone adsorbed on silica gel has been determined as a function of the concn. of acetone adsorbed on the silica. In order to explain the results, it is postulated that acetone is present as multi-molecular layers or 'islands' on the adsorbent, with areas left uncovered until the adsorbent is entirely covered with a multi-molecular layer. This phenomenon is explained in terms of the surface energy of the adsorbent. T. R. ANDREW

2903. **Rapid method for the determination of alkali carbonates and alkali bicarbonates in the presence of one another.** L. Szekeres and E. Bakács-Polgár (Inst. Allg. Chem., Landwirtsch. Univ., Budapest). *Z. anal. Chem.*, 1959, **165** (2), 85–88.—The sample soln. is mixed with ethanol and the CO_3^{2-} content is determined by titration with $BaCl_2$ soln. with phenolphthalein as indicator. A measured excess of $BaCl_2$ soln. is then added and the soln. is heated to decompose the HCO_3^- and form $BaCO_3$. The soln. is cooled and the excess of $BaCl_2$ is back-titrated with standard Na_2CO_3 soln. The accuracy of both determinations is within ± 1%. G. P. COOK

2904. **Determination of germanium.** P. Senise (São Paulo Univ., Brazil). *Selecta Chimica*, 1957, (16), 63–79.—A review is given of methods for the identification of Ge, for its separation from other elements and for its quant. determination, gravimetrically, volumetrically, colorimetrically, polarographically and spectrographically. (86 references.) L. A. O'NEILL

2905. **Spectrochemical determination of germanium in tar ashes.** M. Malinek (Metall. Inst., Czechoslovak Acad. of Sci., Prague). *Anal. Chim. Acta*, 1958, **19** (5), 502–504.—The ash is diluted with Fe_2O_3 to a total Fe_2O_3 content of 50%. Equal wt. of graphite and $CaCO_3$ are added, and the whole thoroughly mixed. Germanium is determined by the internal standard method, the Ge line at 2651·178 A being read against that of Fe at 2645·427 A. Germanium concn. from 0·01 to 1% were determined with a coeff. of variation of 8·6%. T. R. ANDREW

2906. **Photometric determination of tin in metallic lead by means of phenylfluorone.** S. D. Gur'ev and N. F. Sapaeva (Gosudastvennŷĭ Nauch.-Issled. Inst. Tsvet. Metall.). *Zavod. Lab.*, 1958, **24** (10), 1195–1196.—The Sn is first separated from Pb and other elements by means of cupferron from an acid soln. (0·3 to 0·6 *N* HNO_3) and extracted as the Sn - cupferron complex by means of an organic solvent. Tervalent Sb reacts with phenylfluorone and must be oxidised to Sb^{IV} by means of $KMnO_4$. The sample soln. of 20 ml should contain ≯ 500 mg of Pb, since higher concn. affect the extraction of the cupferron complex. *Procedure*—Dissolve 0·5 to 1·0 g of metallic lead (containing ≮ 5 μg of Sn) in 10 ml of HNO_3 (1 : 1) containing 0·5 ml of 50% tartaric acid. Evaporate to dryness on a water bath and dissolve the residue in 0·5 *N* HNO_3 (5 ml for a 0·5-g sample; 10 ml for a 1-g sample). Add 4 to 5 drops of 0·1 *N* $KMnO_4$ and after 5 min. add aq. NH_3 until a ppt. just forms. Add 0·5 *N* HNO_3 dropwise until the ppt. is dissolved, and transfer the soln. to a separating-funnel, washing the beaker with 0·5 *N* HNO_3 (10 ml for a 0·5-g sample, or 20 ml for a 1-g sample; the final volume in the first case should be ≃ 20 ml and in the second case ≃ 40 ml). Add 2 ml or 4 ml (according to the size of the sample) of 1% aq. cupferron soln., and extract with $CHCl_3$ (3 × 5 ml). Discard the aq. fraction and wash the $CHCl_3$ layer with 5 ml of 0·2 *N* HNO_3, evaporate on a water bath, add 5 ml of conc. HNO_3 and evaporate all the $CHCl_3$. Add 5 ml of H_2SO_4 (1 : 1) to the cooled residue, then heat until white fumes of SO_2 are evolved. If the soln. is dark, add a few crystals of NH_4NO_3

and heat to white fumes again. Cool, add a little water, and evaporate to 0·2 to 0·3 ml. Cool again, add 5 ml of 5% tartaric acid, warm and then cool the soln., and transfer it to a 25-ml flask. (If a $PbSO_4$ ppt. is formed, filter the soln. into the flask, washing the ppt. with 2% H_2SO_4.) Add aq. NH_3 until the soln. is alkaline to Congo red paper, followed by 1·6 ml of H_2SO_4 (1:1), 2·5 ml of gelatin soln. (0·5%), then 5 ml of 0·03% ethanolic phenylfluorone soln., acidified with H_2SO_4. Mix well after the addition of each reagent, then make up to 25 ml with water, and after 20 min. measure the extinction of the soln. with a No. 4 light filter and a 30-mm cell. A blank determination must be carried out on the reagents. Results are referred to a calibration curve prepared from a soln. of metallic tin in H_2SO_4. W. ROUBO

2907. **Studies in bivalent chromium salts. VII. Estimation of bivalent tin, sexavalent uranium and quinquevalent vanadium.** J. P. Tandon and R. C. Mehrotra (Chem. Lab. B.R. Coll., Agra Univ., India). *Z. anal. Chem.*, 1958, **164** (3), 314–319 (in English).—In the determination of $SnCl_2$, a standard soln. of Fe^{III} is added and the excess is titrated with $CrSO_4$ soln. (**I**), with KSCN, neutral red or phenosafranine as indicator. Uranyl nitrate is titrated with **I**, with methyl red or *p*-ethoxychrysoidine as indicator, in the presence of excess of H_2SO_4. In an alternative method, U^{VI} is reduced to U^{IV} with **I**. Air is blown into the soln. to oxidise the excess of Cr^{2+}, and the U^{IV} is titrated with $KMnO_4$ or $K_2Cr_2O_7$ soln., with N-phenylanthranilic acid as indicator. Ammonium metavanadate is titrated directly with **I**. Ferric and cupric salts do not interfere. B. B. BAUMINGER

2908. **Quantitative determination of lead by the method of oxidative and inner electrolysis by anodic separation as lead dioxide.** A. Lipčinsky and M. Krstewa (Chem.-Technol. Inst., Sofia, Bulgaria). *Z. anal. Chem.*, 1958, **164** (2), 246–250.—The reaction $PbO_2^{2-} - 2e = PbO_2$ occurs in the following galvanic cell—

$$S_2O_8^{2-} \mid 2SO_4^{2-} \mid PbO_2^{2-} \mid PbO_2 \text{ (Pt)}.$$

Preparation of electrode system—Soak a Soxhlet thimble in collodion soln., dry in air, re-immerse, dry and repeat twice. Before use, steep in water for several hours. Dry, fill with conc. aq. $K_2S_2O_8$ soln. and insert a stout carbon electrode connected to a platinum anode. *Procedure*—Place a known vol. of $Pb(NO_3)_2$ soln. ($\simeq$ 9 mg of Pb) in a 100-ml beaker and add 20% KOH soln. until the ppt. dissolves, with a small excess; dilute with water to 40 ml. Immerse the short-circuited electrodes and stir the $K_2S_2O_8$ soln. by aeration. After a known time, remove the electrodes and dip them into water. Wash the platinum electrode with H_2O, ethanol and diethyl ether, and weigh. At room temp. the recovery is quant. after 30 min., or after 15 min. at 50° to 60°. The deposit dissolves slowly in dil. oxalic acid - HNO_3 soln.
P. D. PARR-RICHARD

2909. **Determination of lead in materials for protective coatings.** R. Montequi and J. Govantes (Inst. 'Alfonso Barba', Madrid, Spain). *Inf. Quim. Anal.*, 1958, **12** (6), 151–156.—The chlorides of Co^{2+}, Mn^{2+}, Zn^{2+} and Ca^{2+} are soluble in 96% ethanol, whereas $PbCl_2$ is practically insoluble, thus providing a convenient method of separation. The $PbCl_2$ is dissolved in hot water and the Cl^- are determined by Volhard's method. *Procedure*—The sample of lead soap is dissolved in benzene (20 to 25 ml), acidified with dil. HCl (1:1) (25 ml) and heated on a water bath for 2 hr. The aq. layer is separated, evaporated to dryness and extracted with 96% ethanol (10 ml). The $PbCl_2$ is filtered off on a sintered glass crucible, washed with ethanol (3 × 5 ml), dissolved in hot water (250 to 300 ml) and determined by Volhard's method. G. H. FOXLEY

2910. **Determination of lead: Report prepared by the Metallic Impurities in Organic Matter Sub-Committee of the Analytical Methods Committee of the Society for Analytical Chemistry.** *Analyst*, 1959, **84**, 127–134.—The residue from a wet- or dry-ashing process is boiled with 5 *N* HCl, the soln. is adjusted to pH 9 to 9·5, KCN soln. is added and, if much Fe is present, hydroxylamine soln. The liquid is extracted repeatedly with a soln. of dithizone in $CHCl_3$ until the final extract is green and the combined extracts are then extracted with dil. HNO_3 (1 + 99). The acid extract is treated with an ammoniacal Na_2SO_3 - KCN soln. and then extracted with 10 ml of $CHCl_3$ containing dithizone, and the extinction of the extract is measured at 520 mμ and referred to a calibration graph. If samples contain much Ca, Mg and PO_4^{3-}, the residue freed from organic matter is adjusted to slight acidity with aq. NH_3 and dil. HCl, warmed with NaI soln., the liberated iodine is removed with $Na_2S_2O_3$, the acidity is adjusted by dilution to *N* in HCl and the liquid is extracted with a soln. of diethylammonium diethyldithiocarbamate in $CHCl_3$. The extract is evaporated with dil. H_2SO_4, fumed with $HClO_4$, boiled after addition of HCl, cooled, treated with ammonium citrate soln. and then treated as previously described, beginning with the adjustment of the pH to 9. Modifications of the details to avoid interference by Bi are described. A. O. JONES

2911. **Titanium diethyldithiocarbamate.** R. C. Rooney (Brit. Cast Iron Res. Ass., Birmingham, England). *Anal. Chim. Acta*, 1958, **19** (5), 428–431.—Quadrivalent Ti forms a complex with Na diethyldithiocarbamate in the pH range 1 to 8. The complex can be extracted into $CHCl_3$ with 100% recovery at pH 2. The extraction is 50% at pH 1·2 and pH 2·4, falling to 10% at pH 4·5, and rising again to 50% at pH 5·3. The extinction of the complex in $CHCl_3$ at 410 mμ was 0·7 for 0·2 mg of Ti per 25 ml in a 1-cm cell. The colour fades rapidly. The reaction is not very suitable for the determination of Ti, owing to the instability of the colour, but can be used to separate Ti from other metals of group 3. T. R. ANDREW

2912. **Spectrophotometric determination of titanium with chromotropic acid.** L. Sommer (Inst. for Anal. Chem., Univ. Brno, Czechoslovakia). *Z. anal. Chem.*, 1958, **164** (3), 299–309.—The reaction mechanism of chromotropic acid with Ti^{IV} at various pH values is discussed. The red $TiOR_2^{6-}$ (**I**) and the orange $TiOR_2^{10-}$ (**II**) complexes are utilised, and a procedure for the determination of Ti in steel is given. Ferric iron is reduced with ascorbic acid at a pH < 1 (*cf.* Koch and Ploum, *Anal. Abstr.*, 1954, **1**, 2944) and the extinction of **I** is measured at 466 to 467 mμ and pH 2·9 to 3·2. An alternative method is described for the determination of Ti with chromotropic acid at pH 5·4 to 6·0, the extinction of **II** being measured at 440 mμ; Fe^{3+} are masked with EDTA, but HCl, HNO_3, F^-, oxalate, PO_4^{2-}, NbO_3^-, WO_4^{2-}, MoO_4^{2-} and VO_3^- interfere in both methods.
B. B. BAUMINGER

2913. **The estimation of bivalent titanium in the presence of the tervalent ion.** H. Martin and J. Stedefeder (Max Planck Inst., Mülheim, W. Germany). *Annalen*, 1958, **618**, 17–23.—Bivalent Ti in ethanolic soln. reacts with $FeCl_3$ (0·1 *N* in 2 *N* aq. HCl) between − 50° and − 70° according to the equation—

$$Ti^{2+} + Fe^{3+} \rightarrow Ti^{3+} + Fe^{2+}.$$

Successful titrations are reported. Total Ti is first determined by conventional methods. Another sample is suspended in dry heptane, cooled to − 70° and treated with ethanol (10 ml). A vol. of 0·1 *N* $FeCl_3$ in 2 *N* HCl equimolar to the total Ti present is added. After slow thawing, 2 *N* HCl (50 ml) is added and the mixture is titrated at 80° with 0·1 *N* $FeCl_3$ to methylene blue. This determines the Ti^{3+} originally present. The procedure is carried out in an atmosphere of N or A with air-free reagents. E. G. CUMMINS

2914. **Colorimetric estimation of zirconium with nitroso-R salt.** V. Gopalakrishna and B. S. V. Raghava Rao (Andhra Univ., Waltair, S. India). *Z. anal. Chem.*, 1958, **164** (4), 384–386 (in English).—Concentrations of Zr as low as $\simeq 0{\cdot}7$ μg per ml can be determined spectrophotometrically at 420 to 460 mμ (10-cm cell) by means of the orange-coloured complex of a zirconyl salt with nitroso-R salt ($\simeq 10^{-3}$ *M* soln.) at pH 2 in the presence of 0·01% of gelatin. Moderate amounts of ThO_2, U_3O_8 or rare-earth oxides, and > 40 μg of TiO_2 or 50 μg of Fe_2O_3 can be tolerated. W. J. BAKER

2915. **Studies on organic reagents for inorganic analysis. V. "*m*-Nitrophenylfluorone" for the spectrophotometric determination of zirconium.** Hirotoshi Sano (Dept. of Chem., Univ. of Tokyo, Hongo, Tokyo). *Talanta*, 1959, **2** (2), 187–190; *Bull. Chem. Soc. Japan*, 1959, **32** (3), 299–303.—The reagent is prepared by dissolving 0·12 g of "*m*-nitrophenylfluorone" [2:3:7-trihydroxy-9-(3-nitrophenyl)fluorone] in 150 ml of ethanol and 1 ml of 5 *N* HCl with warming, and making up to 200 ml with ethanol. To an aliquot (< 10 ml) of test soln. (up to 25 μg of Zr) add acid, if necessary, to bring it to 0·1 *N* with respect to HCl, and water to a vol. of 10 to 12 ml. Add, in order, ethanol (5 ml), *cyclo*hexanol (2·5 ml) and reagent (5 ml). Make up to 25 ml with water, shake, set aside for 1 hr., then measure the extinction at 550 mμ in a 1-cm cell against water. Refer the results to a standard curve, which is linear up to 0·6 p.p.m. and non-linear but reproducible from 0·6 to 10 p.p.m. of Zr. With 0·3 p.p.m. of Zr, no interference was obtained with 2000 p.p.m. of Na^+, K^+, NH_4^+, Mg^{2+}, Cl^-, Br^- or NO_3^-, with 1000 p.p.m. of Ca^{2+}, Ba^{2+}, Sr^{2+}, Hg^{2+} or SO_4^{2-}, with 500 p.p.m. of La^{3+}, Nd^{3+}, Ce^{3+}, Cd^{2+} or Zn^{2+}, or with 100 p.p.m. of Al^{3+} or Th^{4+}. Interference was observed with 15 p.p.m. or less of Fe, Ti, Ge, Sn, As, Sb, F^-, HPO_4^{2-} and $C_2O_4^{2-}$. The sensitivity is 50 times that of the alizarin red S method, and five times that of the quercetin method. The procedure is suitable for micro-determinations. R. E. ESSERY

2916. **Determination of zirconium in high-temperature alloys.** K. L. Cheng (Utica Metals Div., Kelsey-Hayes Co., Utica, N.Y., U.S.A.). *Talanta*, 1959, **2** (2), 186–187.—Application is made of the previously reported colour reaction of Zr and Hf with xylenol orange (*cf. Talanta*, 1959, **2**, 61). Dissolve the alloy (1 g) with conc. HCl (30 ml) and conc. HNO_3 (10 ml) with heating. Add 30 ml of H_2SO_4 (1:1) and fume for a few minutes. Cool, dilute to 100 ml, and remove Ni, Co, etc., with a mercury cathode at 25 amp. till only a faint thiocyanate reaction for Mo is given. Add 10 *N* NaOH to precipitate Zr and Ti, and digest for 15 min. on a hot-plate. Filter (Whatman No. 42), wash thrice with hot water, dissolve the ppt. in hot 0·8 *N* $HClO_4$, cool, and make up to 250 ml with 0·8 *N* $HClO_4$. Determine Zr in a suitable aliquot as directed in the original method. Results agree well with those by the gravimetric phosphate method. R. E. ESSERY

2917. **The analysis of zirconium - thorium binary alloys.** G. W. C. Milner and J. W. Edwards (Anal. Chem. Group, A.E.R.E., Harwell, England). *Anal. Chim. Acta*, 1959, **20** (1), 31–37.—Total Zr and Th is determined by complexometric titration, the Th is then masked with $(NH_4)_2SO_4$ and the liberated EDTA is determined by titration with standard bismuth soln. *Procedure*—Dissolve the sample in conc. HNO_3 (10 ml) containing 40% HF (4 to 5 drops). Add 60% $HClO_4$ (20 ml) and evaporate to 10 ml. Add 5 *N* HNO_3 (25 ml) and dilute to 100 ml. To a suitable aliquot add excess of 0·02 *M* EDTA (disodium salt) and dilute to 150 ml. Add 0·1% xylenol orange soln. (1 drop) and adjust the pH to 2·6 to 2·8 with aq. NH_3. Add 50% (w/v) $Be(NO_3)_2$ soln. (3 ml) and titrate with 0·02 *M* $Bi(NO_3)_3$. Add $(NH_4)_2SO_4$ (2 to 3 g), adjust the pH to 1·2 to 1·3 with conc. HNO_3 and titrate again with 0·02 *M* $Bi(NO_3)_3$. For alloys containing < 20% of Th, a preliminary separation of Th by co-pptn. with lanthanum fluoride is necessary, and for samples containing < 5% of Zr a preliminary separation of Zr with *p*-bromomandelic acid. The method is applicable to Zr - Th binary alloys containing 1 to 99% of either component. W. T. CARTER

2918. **Spectrographic analysis of Zircaloys.** J. A. Wheat (E. I. du Pont de Nemours & Co., Aiken, S. C., U.S.A.). *Appl. Spectroscopy*, 1958, **12** (5), 152–153.—Tin (0·2 to 1·8%), Fe (0·08 to 0·3%), Cr (0·01 to 0·12%) and Ni (0·01 to 0·08%) can be determined in Zircaloys by the point-to-plane spark spectrographic method. Coefficients of variation are 4% for Sn, Cr and Ni, and 6% for Fe. Under suitable conditions the method can be applied to rolled sheet and thin-walled tubing, as well as to large blocks. K. A. PROCTOR

2919. **The thorium - APANS [thoron] complex. A spectrophotometric study.** A. R. Palmer (Austral. Atomic Energy Comm. Res. Lab., Sydney, Australia). *Anal. Chim. Acta*, 1958, **19** (5), 458–461.—Thoron (**I**) reacts with Th^{4+} to give a complex in the ratio 2 molecules of (**I**) to 1 atom of Th. T. R. ANDREW

2920. **Determination of thorium oxide in thoriated tungsten wire.** D. H. Wilkins (Res. Lab., General Electric Co., New York, U.S.A.). *Anal. Chim. Acta*, 1958, **19** (5), 440–441.—Thorium oxide in tungsten wire is determined by dissolving the sample in a mixture of HF and HNO_3, evaporating to strong fumes with H_2SO_4 and, after quadruple pptn. of $Th(OH)_4$ with KOH (to ensure separation from W), dissolution in HCl, addition of excess of EDTA (disodium salt) at pH 4·5 and back-titration with standard Cu^{2+} soln. T. R. ANDREW

2921. **Rapid determination of nitrogen by distillation in superheated steam and simultaneous titration.** P. Navellier (Munic. Lab., Paris). *Fruits d'Outre Mer*, 1958, **13** (9–10), 410–411.—Nitrogen

is determined in samples containing from 5 to 100 mg, after conversion into NH_3, by passing steam at 250° to 300° through the sample in a specially fitted Kjeldahl flask. The vapour is condensed into the titrating beaker. One operator can carry out 100 determinations per day.

M. D. ANDERSON

2922. **Determination of hydrazine and hydrazine derivatives using bromine chloride as standard solution.** E. Schulek and K. Burger (Inst. for Inorg. and Anal. Chem., L. Eötvös Univ., Budapest, Hungary). *Talanta*, 1958, **1** (4), 344–350.—The stable standard soln. of BrCl (*Anal. Abstr.*, 1959, **6**, 2222) has been employed for the titration of hydrazine and its derivatives, with 0·2% ethanolic *p*-ethoxychrysoidine as indicator. The substance (0·3 g) is dissolved in water and made up to 100 ml. Aliquots (5 to 10 ml) of this soln. are diluted to 30 to 50 ml with water, treated with 2 to 10 ml of 20% HCl or 10 to 20 ml of 2 *N* H_2SO_4 and 1 drop of indicator, and titrated with 0·1 *N* BrCl to a yellow end-point. Results are not affected by the degree of acidity or the nature of the acid. Results for hydrazine sulphate, phenylhydrazine hydrochloride, semicarbazide hydrochloride and isoniazid in amounts between 12 and 37 mg show good recoveries.

R. E. ESSERY

2923. **Polarographic determination of hydroxylamine and nitroethane.** P. Vetešník and S. Havel (Inst. Org. Chem., High-School of Chem. Technol., Prague). *Chem. Průmysl*, 1958, **8** (10), 526–527.—The polarographic reduction of 0·001 *M* soln. of nitroethane (**I**) and hydroxylamine (**II**) and of their mixtures has been studied. In Britton-Robinson buffer soln. (pH 6 to 7), **II** yields one and **I** two polarographic waves. The wave of **II** and the first wave of **I** coincide, but the height of the wave corresponds quant. to the total amount of both compounds. By constructing calibration curves, the amounts of both compounds in a mixture can be determined from the difference of the wave heights.

J. ZÝKA

2924. **Gravimetric modification of the Wilson method for the determination of phosphorus.** C. H. Perrin (Canada Packers Ltd., Toronto). *J. Ass. Off. Agric. Chem.*, 1958, **41** (4), 758–763.—Organic matter in the sample is destroyed by treatment with HNO_3 and $HClO_4$ or HCl. The slightly acid soln. is treated with a citric acid - molybdic acid soln. (specified) followed by a quinoline hydrochloride reagent (specified). The ppt. quinolinium molybdophosphate is washed with water and dried to constant wt. at 250°, when it contains 3·2074% of P_2O_5. Results are accurate. A simplified procedure for routine determinations is described.

A. A. ELDRIDGE

2925. **Ferrimetric determination of phosphite: catalysis by palladium chloride.** G. Gopala Rao and G. Somidevamma (Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1958, **164** (4), 391–394 (in English).—A volumetric determination of phosphite, in 50 ml of soln., can be made by oxidation to PO_4^{2-} with an H_2SO_4 soln. of $(NH_4)_2SO_4.Fe_2(SO_4)_3$ (0·025 *M*) at 100° for 10 min. in the presence of 1 ml of 0·1% aq. $PdCl_2$ soln. as catalyst. After cooling the soln. and adding conc. H_3PO_4 (5 ml), the Fe^{2+} are titrated with $NaVO_3$ soln. ($\simeq$ 0·05 *N*), with diphenylbenzidine as indicator. The concn. of H_2SO_4 during the oxidation should be 0·1 to 0·2 *N*. For phosphite concn. of 0·1 to 0·3 m*M*, the error is $\pm$ 0·1%.

W. J. BAKER

2926. **Amperometric determination of arsenic and selenium by the direct iodide method.** O. A. Songina and A. P. Volloshnikova (Kazakh State Univ.). *Zavod. Lab.*, 1958, **24** (11), 1331–1336.—With a rotating platinum electrode and a mercury-iodide reference electrode, As^V in 10 *N* HCl is titrated amperometrically at + 0·2 V with KI soln. using the cathodic reduction wave of iodine. No interference is caused by Fe in the strongly acid medium, nor by Pb, Ag, Hg and Tl, since these form insol. chlorides or stable chloride complexes. The presence of Fe reduces interference due to Sb. The interference of Cu is prevented by reduction to the metal with cadmium amalgam, which does not reduce As^V. For the determination of Se^{IV}, the anodic oxidation current of excess of I^- is used. The methods are applicable to the determination of 0·01 to 0·1-mg amounts of As and Se.

G. S. SMITH

2927. **Bismuthiol I as an analytical reagent. Estimation of bismuth and palladium.** A. K. Majumdar and M. M. Chakrabartty (Jadavpur Univ., Calcutta, India). *Z. anal. Chem.*, 1959, **165** (2), 100–105 (in English).—Bismuth and Pd are determined volumetrically after pptn. as the complex with Bismuthiol I (2:5-dimercapto-1:3:4-thiadiazole) (**I**). From 0·1 *N* HCl soln. they are separated from Fe^{3+}, Al, Cr, Ce^{3+}, Zr, Ti, Zn, Th, UO_2^{2+}, Be, Mg, Mn, Co, Ni and alkaline-, rare-earth and alkali metals. For the determination of Bi, the Bi - **I** complex is dissolved in EDTA, the excess of which is titrated with $MgSO_4$ soln., with Eriochrome black T as indicator. The Pd - **I** complex is dissolved in KCN soln., the excess of which is back-titrated with $AgNO_3$ soln. A mixture of tartrate or citrate and EDTA is useful for the separation of Pd from many other ions. The accuracy of each determination is within $\pm$ 1% at the 10-mg level. Bivalent Sn, Sb^{3+}, Fe^{3+}, F^-, VO_3^-, PO_4^{3-} AsO_4^{3-}, and CrO_4^{2-} interfere in the determination of Bi, and Sn^{2+}, Pt^{4+} and CN^- in the determination of Pd.

G. P. COOK

2928. **Potentiometric studies on bismuth ferrocyanide and the determination of bismuth.** Ram Sahai Saxena and C. S. Bhatnagar (Gov. Coll., Kota, India). *Z. anal. Chem.*, 1959, **165** (2), 95–99 (in English).—The reaction between $Bi(NO_3)_3$ and the ferrocyanides of alkali metals was studied potentiometrically with a platinum indicator electrode and an S.C.E. The end-point obtained from the maximum on the d*E* *vs.* d*V* curve corresponds to the formation and complete pptn. of a compound with the formula $BiMFe(CN)_6$, where M represents an alkali metal. The curves are reproducible and provide a means of determining Bi to within $\pm$ 0·5% at the 0·01 to 0·2 *M* levels. The effects of ethanol and electrolytes on the course of the reaction were studied.

G. P. COOK

2929. **Chemical analysis and separation by the extraction method. VI. Spectrophotometric determination of vanadium with phenylhydrazine.** Sakujiro Yamamoto (Fac. of Liberal Arts, Shimane Univ., Matsue). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1958, **79** (11), 1309–1314.—The extraction of V from an HCl soln. with ether in the presence of KSCN and phenylhydrazine (**I**) is not quant. but is reproducible for amounts from 1 to 30 μg of V under given conditions. The extinction of the ethereal soln. at 400 mμ is proportional to concn. of V from 5 to 30 μg. The optimum conditions for the extraction are as follows. The sample

soln. (5 to 10 ml) is mixed with 6 *N* HCl (0·1 ml), KSCN soln. (10%) (0·5 ml) and **I** [in dil. (1:2, v/v) acetic acid] (2%, w/v, pH 1·3) (0·6 ml) and shaken with an equal vol. of ether. K. SAITO

2930. **The separation of molybdenum from vanadium by anion exchange as the thioglycollate complex and the photometric determination of vanadium with thioglycollic acid.** M. Ziegler and W. Rittner (Inorg. Chem. Inst., Univ. Göttingen, Germany). *Z. anal. Chem.*, 1958, **164** (3), 310–313.—*Procedure*—Dowex 1 (SO_4^{2-}) (4 ml) is packed into a tube (15 cm × 1 cm) and washed until neutral. To an alkaline soln. of MoO_4^{2-} and VO_4^{3-} is added 10 ml of 8% thioglycollic acid, and the soln. is adjusted to pH 1 to 1·5 with 0·1 *N* H_2SO_4, then added to the column, which is washed with 0·1 *N* H_2SO_4. Vanadium passes through the column and Mo is adsorbed. Elution of the Mo is effected with 100 to 150 ml of conc. aq. NH_3 - water (1:7) containing 3% of $(NH_4)_2S_2O_8$, and, after boiling the eluate to decompose the persulphate and buffering with Na acetate, Mo is determined gravimetrically with 8-hydroxyquinoline (*cf.* Geilmann and Weibke, *Z. anorg. Chem.*, 1931, **199**, 347), with a recovery of 99 to 100%. The V-containing soln. is evaporated to about 70 ml, transferred to a flask containing 10 ml of 8% thioglycollic acid and 10 ml of buffer soln. (2·6% of ammonium acetate, 11% of Na acetate and 0·6% of glacial acetic acid) (pH 6·7) and made up to 100 ml. The extinction is measured at 620 mμ. The results are tabulated.

B. B. BAUMINGER

2931. **Separation and determination of niobium and tantalum by partition chromatography.** I. A. P. Scott and R. J. Magee (Chem. Dept., The Queen's Univ., Belfast, N. Ireland). *Talanta*, 1958, **1** (4), 329–333.—Separation is achieved by means of a mixture of 2·5 ml of 4% HF with 50 ml of *iso*butyl methyl ketone. This is a two-phase mixture, and Nb moves with the acid front and Ta with the ketone front. Place the mixture in the trough of the chromatography tank at least 1 hr. before commencing the separation. With Whatman No. 1 paper, mark lines at 2·5, 4 and 6 cm from the top, bend the paper along the 2·5-cm line and load the soln. of the metals in HF (0·1 ml) along the 6-cm line. Insert the paper into the tank while the spots are still wet, and develop by descending chromatography until the acid front reaches the 10-cm mark and the ketone front the 15-cm mark. This takes 3 hr. Dry the paper in air, and spray with 5 *N* aq. NH_3 and then with a 5% soln. of 8-hydroxyquinoline in methanol - $CHCl_3$ - water (12:12:1). Dry at 120° for 30 min., wash with water to remove excess of 8-hydroxyquinoline, then dry for 1 hr. Under u.v. light Nb is shown as a brilliant yellow band (R_F 0·60), while Ta is detected by the naked eye (R_F 0·90). The lower limit of detection for both metals is 20 μg. For the determination, the bands after detection are cut from the paper and extracted with 10 ml of hot 2 *N* HCl. For Nb, the soln. is adjusted with aq. NH_3 to pH 8·5 and the 8-hydroxyquinolinate is extracted with $CHCl_3$ (3 × 3 ml), the extract dried with Na_2SO_4, made up to 10 ml, and the Nb determined spectrophotometrically at 385 mμ. For Ta, the extraction is at pH 10, and the determination at 390 mμ. Results are referred to calibration curves. The procedure for the analysis of a stainless steel is described. R. E. ESSERY

2932. **Determination of tantalum by differential spectrophotometry.** B. M. Dobkina and T. M. Malyutina (State Sci. Res. Inst. of Rare Metals). *Zavod. Lab.*, 1958, **24** (11), 1336–1343.—The sample (0·1 to 0·2 g) of tantalum concentrate, containing much Nb and some Ti, is fused with 4 to 8 g of $K_2S_2O_7$ with the dropwise addition of conc. H_2SO_4 to give a transparent melt, which is then dissolved in 40 to 50 ml of 4% ammonium oxalate soln. An aliquot (2 to 3 ml) of the soln. obtained by dilution to 100 or 200 ml with 4% ammonium oxalate soln. is diluted further, if necessary, with the same reagent, so that the amount of ammonium oxalate in 10 ml is 120 mg whilst that of $K_2S_2O_7$ is 50 to 100 mg. Carefully measured amounts of 8 *N* HCl (5 ml) and 20% pyrogallol soln. (2 ml) are added successively, followed by stirring, and the soln. is diluted to 25 ml. The extinction in a 1-cm cell is measured at 325 mμ with a reference soln. containing Ta_2O_5 (0·7 or 0·8 mg) and all the reagents. With a ratio of Nb_2O_5 to Ta_2O_5 of 3:1 the error in determining Ta is ≃ 0·5% of the content. With a ratio of TiO_2 to Ta_2O_5 of 1:6 the error is also ≃ 0·5% of the content of Ta. G. S. SMITH

2933. **Polarography of gases. Quantitative studies of oxygen and sulphur dioxide.** D. T. Sawyer, R. S. George and R. C. Rhodes (Univ. of California, Riverside, U.S.A.). *Anal. Chem.*, 1959, **31** (1), 2–5.—The polarographic behaviour of a large number of gaseous materials has been investigated and $E_{\frac{1}{2}}$ values and diffusion currents were determined in many cases. A plastic membrane was used as the diffusion barrier for the working electrode. The method can be used over the concn. range 0·01% to 100% (by vol.), and a quant. procedure has been developed for determining O and SO_2, both in the gaseous phase and in soln.

K. A. PROCTOR

2934. **Mass spectrometry of impurities in liquid oxygen.** Keinosuke Suzuki, Shun Araki and Shizo Hirano (Inst. of Techno-anal. Chem., Fac. of Engng, Tokyo Univ., Hongo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1958, **61** (12), 1556–1560.—Liquid oxygen (100 to 200 g) is caused to evaporate in a 400-ml metal cylinder, cooled with liquid oxygen, at a rate of < 2 litres per min. and the gas is passed through two glass traps (capacity 30 to 45 ml) cooled with liquid oxygen. Impurities (< 600 p.p.m.) thus captured were submitted to mass spectrometry. The amount of the sample was measured with a flow meter. Hitherto unidentified impurities including N_2O (≃ 100 p.p.m.), C_2 to C_5 hydrocarbons (20 to 500 p.p.m.), Xe, Kr and CO_2 were detected by means of the peaks *e/m* 22 (CO_2), 84 (Kr), 132 (Xe), 43, 29 (propane, butane and pentane), 27 (olefins), 30 (N_2O and ethane), 44 (N_2O, C_3H_8 and CO_2) and 26 (acetylene).

K. SAITO

2935. **A spectrophotometric method of determining sulphuric acid concentration.** E. Zimmerman and W. W. Brandt (Dept. of Chem., Purdue Univ., Lafayette, Ind., U.S.A.). *Talanta*, 1958, **1** (4), 374–376.—The absorption spectra of quinalizarin (**I**) in H_2SO_4 of various concn. show maxima at 537, 575 and 605 mμ, and an isosbestic point at 585 mμ. The addition of H_2O to the system increases the extinction at 537 and 575 mμ, and decreases that at 605 mμ. The ratio of the extinctions at 535 and 630 mμ gives the best curve, and values of this ratio, plotted against

concn. of H_2SO_4, give a smooth curve between 85 and 99% H_2SO_4, which reaches a minimum at between 99 and 99·5% and then rises sharply. After such a calibration curve has been prepared, the determination is made by dissolving a little **I** in 3 to 5 ml of the sample, reading the extinctions at 535 and 630 mμ, calculating the ratio, and referring it to the curve. Results are accurate to within ± 0·3%. Borate interferes, causing high results. If the sample is coloured, the extinctions are determined before and after the addition of **I**, and the ratio of the increases in the extinctions is calculated and referred to the curve.

R. E. ESSERY

2936. **Successive determination of sulphuric acid and sulphonic acids by titration in non-aqueous solvents.** E. A. Gribova and E. S. Levin (K. E. Voroshilov Inst. of Org. Intermediates and Dyestuffs). *Zavod. Lab.*, 1958, **24** (11), 1356–1358.—The acids in dry methyl cyanide or acetone soln. are titrated potentiometrically with 0·1 *N* diphenylguanidine in ethanol containing 10 to 15% of glycerol to prevent crystallisation at the jet of the burette. The first potential jump corresponds to the completion of titration of sulphonic acids and the formation of HSO_4^- from H_2SO_4. The second jump corresponds to complete neutralisation of H_2SO_4, but in the presence of the organic solvent it is not well defined. Dilution with twice the vol. of water permits a good end-point to be obtained.

G. S. SMITH

2937. **Fluorimetric determination of sulphate ions and spectrophotometric determination of thorium by means of a trihydroxyfluorone derivative.** V. A. Nazarenko and M. B. Shustova (Inst. of Gen. and Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1958, **24** (11), 1344–1346.—In acid medium (pH preferably between 4 and 5), 2:3:7-trihydroxy-9-(*o*-hydroxyphenyl)-6-fluorone (**I**) gives with salts of Th a bright red soln. or, with high contents of Th, a ppt. At pH 4·4 and 530 mμ the mol. extinction coeff. of the complex measured over the concn. range 0·3 to 1·0 × 10^{-5} *M* is 26,000. With soln. 3 × 10^{-5} *M* in **I**, and containing 20% of ethanol and 0·02% of gelatin, Beer's law is obeyed over the concn. range 0·5 to 9 × 10^{-6} *M*. No interference is caused by ≯ 100 μg of La, Ce^{3+} and Y in 25 ml of soln. Free **I** fluoresces in u.v. light but the complex with Th does not. Since Th combines preferentially with SO_4^{2-}, a suitable choice of conditions permits the determination of SO_4^{2-}. To detect microgram amounts of SO_4^{2-}, the soln. (1 ml) is mixed with 0·5 ml of ethanol and 0·5 ml of 2 × 10^{-4} *M* $Th(NO_3)_4$ followed, after 5 min., by 0·2 ml of 5 × 10^{-5} *M* **I** (prepared by dissolving 33·6 mg of **I** in 100 ml of 96% ethanol and 0·1 ml of 6 *N* HCl by gentle heating, and then diluting 5 ml to 100 ml with ethanol). The fluorescence is observed in the filtered light of a mercury lamp. To determine SO_4^{2-}, comparison is made with a series of standards containing 0, 0·25, 0·5, 1·0, 1·5 and 2·0 μg of SO_4^{2-}. In the absence of SO_4^{2-}, the soln. is pink and does not fluoresce. In its presence, a bright greenish-yellow fluorescence appears. Similar reactions are given by PO_4^{3-} and F^-, but the sensitivity is lower. The method is applied to the determination of SO_4^{2-} in pure germanium dioxide.

G. S. SMITH

2938. **Analytical applications of ferricyanide oxidations catalysed by osmium tetroxide. II. Direct determination of sulphur compounds with potassium ferricyanide.** F. Solymosi and A. Varga (Inst. Inorg. and Anal. Chem., Univ., Szeged, Hungary). *Magyar Kém. Foly.*, 1958, **64** (11), 443–447.—Sulphites, sulphides, thiosulphates and tetrathionates are titrated with ferricyanide in strongly alkaline soln. at 50° to 60°, with OsO_4 as catalyst. Under these conditions, atmospheric oxidation is negligible. The error is < 0·2%.

G. SZABO

2939. **Voltammetry of thiocyanate ion at the stationary platinum electrode.** M. M. Nicholson (Humble Oil & Refining Co., Baytown, Tex., U.S.A.). *Anal. Chem.*, 1959, **31** (1), 128–132.—Thiocyanate ions in pyridine - pyridinium ion soln. can be determined in the presence of excess of Cl^- by an anodic voltammetric procedure with a stationary platinum electrode. For 0·1 to 4 millimoles per litre, the ratio of max. current to concn. may be determined with an average deviation of ± 1%. The effects of CN^-, Br^- and I^- have been studied.

K. A. PROCTOR

2940. **Determination of selenium with ferrous sulphate.** A. M. Scherhaufer (Inst. Inorg. Anal. Chem., Tech. Coll., Vienna, Austria). *Z. anal. Chem.*, 1958, **164** (3), 327–335.—Methods for the gravimetric and volumetric determination of SeO_3^{2-} and SeO_4^{2-} are described. Selenite in the presence of SeO_4^{2-} is determined by the method of Norris and Fay (*Amer. Chem. J.*, 1896, **18**, 703). The selenite soln. is diluted with H_2O, acidified with HCl, and cooled to 0° to 5°; a slight excess of $Na_2S_2O_3$ soln. is added and the excess is back-titrated with iodine soln. In a second sample the total Se is determined with $FeSO_4$. An alternative method is also given by which the determination of SeO_3^{2-} and SeO_4^{2-} can be carried out on a single sample. The apparatus is illustrated. Selenite in the presence of TeO_3^{2-} can be determined gravimetrically and TeO_3^{2-} does not interfere. In the volumetric determination of Se, TeO_3^{2-} are pptd. as TeO_2 and the method of Browning and Flint (*Amer. J. Sci.*, 1909, **28**, 112, 117) is used. The results are tabulated.

B. B. BAUMINGER

2941. **Photometric determination of selenium.** H. Bode and H. Mösenthin (Anorg.-chem. Inst., d. Tech. Hochschule, Hannover). *Z. anal. Chem.*, 1958, **164** (2), 232–241.—Small amounts (5 to 100 μg) of Se are determined indirectly by pptn. as Cu_2Se; Cu in the ppt. is measured photometrically. *Procedure*—To each 10 ml of neutral Se^{IV} soln. add 5 ml of buffer soln. (5 g of citric acid and 400 mg of $CuSO_4.5H_2O$ adjusted to pH 4·5 and to 100 ml with aq. NH_3) and 1 ml of aq. hydroxylamine sulphate soln. (10%), and place a small funnel in the neck of the flask. Boil for 3 min., cool and rinse the funnel and walls of the flask with a few ml of methanol. Filter the contents through a micro filter-stick (porosity 4), wash the flask and stick with 3 ml of H_2O, then several times with methanol, and finally with 1 to 3 ml of H_2O. Return the stick to the flask, warm with 1 ml of HNO_3 ($d = 1·4$), remove the stick and wash it with H_2O into the flask; the final vol. of the soln. should be ≮ 25 ml. Shake the soln. with 25 ml of diethylammonium diethyldithiocarbamate soln. (0·04% in CCl_4) and filter the organic phase through a dry paper into a 1-cm cell (for 5 to 40 μg of Se). Measure the extinction at 436 mμ *vs.* CCl_4. Silver and Pd interfere; Au, Bi, Hg, Mo, Pt, Rh, Sn and Te may be present in small amounts; As^{3+}, Cd^{2+}, Co^{2+}, Fe^{2+}, Mn^{2+}, Ni^{2+}, Pb^{2+}, Sb^{3+}, Tl^+ and Zn^{2+} do not interfere. The total analysis takes 20 to 30 min.

P. D. PARR-RICHARD

2942. **Fluorimetric determination of tellurium with rhodamine B.** D. P. Shcherbov and A. I. Ivankova (Kazakh Inst. of Mineral Raw Materials). *Zavod. Lab.*, 1958, **24** (11), 1346–1349.—The behaviour of a number of metal ions under the conditions for the fluorimetric determination of Te with rhodamine B (*cf. Anal. Abstr.*, 1959, **6**, 1658) is studied. G. S. SMITH

2943. **Complexometric determination of copper, nickel and iron in tungsten alloys.** E. Lassner, R. Scharf and P. L. Reiser (Versuchanst. der Metalwerk Plansee A.-G. Reutte, Tirol, Austria). *Z. anal. Chem.*, 1959, **165** (2), 88–94.—The sample is dissolved in a mixture of HF and HNO_3, evaporated to dryness, and heated to fuming with conc. H_2SO_4. The residue is dissolved in a mixture of NaOH and tartaric acid, the acid acting as a mask for W in the titrations. The soln. is diluted with H_2O and the pH is adjusted to 3·5 to 3·8. Three titrations are employed; in each an excess of EDTA is added and 1-(2-pyridylazo)-1-2-naphthol used as indicator. The first titration determines all three elements and $CuSO_4$ soln. is used to back-titrate the excess of EDTA. In the second titration the Cu and Ni are determined, the Fe being masked by addition of NH_4F; $CuSO_4$ soln. is used as titrant. Only Ni is determined in the last titration, the Cu and Fe being masked by addition of $Na_2S_2O_3$ and NH_4F; $NiSO_4$ soln. is used as titrant. The relative error ranges from 0·2 to 1·5%. G. P. COOK

2944. **Cerimetric determination of uranium(IV). Use of triphenylmethane dyes as internal indicators.** V. Panduranga Rao and G. Gopala Rao (Andhra Univ., Waltair, S. India). *Talanta*, 1958, **1** (4), 355–358.—The use of Erioglaucine A, Erio green B and xylene cyanol FF as internal indicators in the titration of U^{IV} with $Ce(SO_4)_2$ is studied. The dyes are used as 0·1% soln., 0·5 ml per titration, and the colour change from yellow to orange-red at the end-point is rapid and reversible at room temp. within an acidity range of 0·5 to 8·0 *N* with respect to H_2SO_4. With 0·05 *N* $Ce(SO_4)_2$, the indicator correction is negligible, but a correction of 0·04 ml is required with a 0·1 *N* soln. Of the three dyes, Erioglaucine A is preferable, as it gives a more sensitive colour change. Recoveries of from 0·0250 to 0·4488 millimoles of U^{IV} with the three dyes were satisfactory. The indicators can be used successfully in HCl soln. and for titrations in $HClO_4$ media with $(NH_4)_2[Ce(NO_3)_6]$ soln. They cannot be used for titrations with $(NH_4)_2[Ce(NO_3)_6]$ soln. in HNO_3 media. R. E. ESSERY

2945. **Complexometric determination of uranium(VI) with EDTA and PAN indicator.** E. Lassner and R. Scharf (Metallwerk Plansee A.-G., Reutte, Tirol, Austria). *Z. anal. Chem.*, 1958, **164** (4), 398–402.—Milligram amounts of U^{VI} in H_2O - *iso*propyl alcohol (1:2) can be titrated at 80° to 90° with EDTA, the end-point with the indicator PAN [1-(2-pyridylazo)-2-naphthol] being from intense red to pale yellow. The sample soln. should be adjusted to pH 2·2 (with HNO_3) and then buffered to pH 4·4 (with hexamine) before dilution with *iso*propyl alcohol. When Cu or Ni is present, the titration is made as usual, and enough Na_2PO_4 soln. is added to precipitate all the UO_2^{2+}; the ppt. and soln. are then titrated with a standard soln. of Cu to a violet end-point. The vol. of Cu soln. used, when converted into the equivalent of EDTA, represents the content of U. The max. absolute error is ± 0·3 mg, and ≯ 100 mg of U per 300 ml of soln. should be present. W. J. BAKER

2946. **Polarographic reduction of sexavalent uranium in sodium triphosphate. I. General studies.** P. R. Subbaraman, N. R. Joshi and J. Gupta (Nat. Chem. Lab., Poona, India). *Anal. Chim. Acta*, 1959, **20** (1), 89–95.—The uranyl ion in alkaline soln. containing sodium triphosphate undergoes an irreversible one-electron reduction with an $E_{\frac{1}{4}}$ of −1·07 V *vs.* the S.C.E. at pH 9. Indifferent electrolytes shift the $E_{\frac{1}{4}}$ to more positive potentials, and capillary-active substances have the opposite effect. A well-defined wave suitable for analytical purposes is obtained in the presence of 0·01% of camphor. W. T. CARTER

2947. **Colorimetric uranium determination with arsenazo.** J. S. Fritz and M. Johnson-Richard (Iowa State Coll., Ames, U.S.A.). *Anal. Chim. Acta*, 1959, **20** (2), 164–171.—The spectral curves of arsenazo and the U - arsenazo complex at various pH values indicate that the absorption max. at 595 mμ and a pH value between 7·0 and 8·0 represent the best conditions for the determination of U. The ratio of U to arsenazo in the complex is 1:1 and, at 595 mμ, ϵ = 22,900. Many other metals form complexes, but interference is limited by the addition of EDTA, the excess of which must not exceed 0·005 millimole per 50 ml to avoid interference with the determination of U. Tartrate is used to mask Mo^{6+}, W^{6+} and Ti^{4+}, sulphosalicylate for Al^{3+}, and 3-mercaptopropane-1:2-diol for Sb, Bi and Cu. Results 2·4% low are produced in the presence of a 0·2 *M* concn. of SO_4^{2-}, and PO_4^{3-} must be absent. If Th^{4+} are present, procedure (*ii*) must be used. *Procedure* (*i*)—Take an aliquot of the soln. that contains 5 to 500 μg of U and adjust the pH to a value of 2 to 3. Add sufficient 0·01 *M* EDTA to complex foreign metals and add ≯ 0·5 ml in excess. Add 2 ml of a 0·001 *M* soln. of purified arsenazo and 5 ml of triethanolamine buffer (equal vol. of *M* triethanolamine and 0·5 *N* HNO_3) and dilute to 40 ml. Adjust to pH 7·5 with dil. HNO_3 or aq. NH_3 and dilute to 50 ml. Measure the extinction in a 1-ml cell at 595 mμ and interpret the results by means of a calibration graph. *Procedure* (*ii*)—To a soln. aliquot containing ≯ 0·08 millimole of foreign metal add 2 ml of 0·05 *M* EDTA and 1 ml of pyridine. Dilute to 20 ml, adjust the pH to a value between 7 and 8, set aside for 15 to 30 min., add 3 ml of 2% Na diethyldithiocarbamate soln. and extract with 25 ml of $CHCl_3$. Add a further 1 ml of dithiocarbamate soln. and extract with 15 ml of $CHCl_3$. Combine the $CHCl_3$ extracts and extract them with 50 ml and 25 ml of 0·02% Na_2CO_3 soln. To the combined aq. Na_2CO_3 extracts add 1 ml of conc. $HClO_4$, 5 ml of triethanolamine buffer and 5 ml of 0·001 *M* arsenazo, adjust the pH to 7·5, dilute to 200 ml and determine the extinction at 595 mμ. H. N. S.

2948. **Spectrophotometric determination of uranium with neothorone [arsenazo].** Shozo Shibata and Teiichi Matsumae (Gov. Ind. Res. Inst., Nagoya Kita-ku). *Bull. Chem. Soc. Japan*, 1959, **32** (3), 279–282 (in English).—The method described enables 5 to 40 μg of uranium in 25 ml of soln. to be determined easily and accurately. Interference by small amounts of Al, Zn, Pb, Th and Fe is avoided by the use of EDTA. I. JONES

2949. **Spectrophotometric determination of uranium. Application of the method to the analysis of leach solutions and monazite.** K. S. Koppikar, V. G. Korgaonkar and T. K. S. Murthy (Atomic Energy Est., Trombay, Bombay, India). *Anal. Chim. Acta*, 1959, **20** (4), 366–371.—A study of the reaction of UO_2^{2+} with SCN^- indicates conditions in which UO_2^{2+} can be determined without a preliminary separation (*cf.* Clinch and Guy, *Anal. Abstr.*, 1958, **5**, 2193). Of a number of metals tested, only V^{5+}, Bi^{3+} and Mo^{6+} offer serious interference with the method, and it may be applied directly to some uranium-yielding ores, including monazite, that are free from these metals. Oxalate and F^- also interfere. *Procedure*—Treat the ore with H_2SO_4 and to an aliquot of the leach soln., containing 0·4 to 0·9 mg of U calculated as U_3O_8, add 5 ml of 5% ascorbic acid soln. (to reduce Fe^{3+}) and 2·5 ml of 2·5 *M* NH_4SCN. Dilute to $\simeq$ 25 ml, adjust the pH to $\simeq$ 1·5 and extract with 25 ml of a solvent (prepared by diluting 100 ml of tributyl phosphate to 1 litre with CCl_4 and washing the mixture three times with 250-ml portions of 10% w/v NaOH soln. and then with water until free from alkali). Filter the extract through glass wool previously washed with HCl and dried, and determine the extinction at 365 mμ. H. N. S.

2950. **Extraction and spectrometric determination of uranium with thenoyltrifluoroacetone.** Shripad M. Khopkar and Anil K. De (Jadavpur Univ., Calcutta, India). *Chem. & Ind*, 1959, (9), 291.—Small amounts of U are completely and rapidly extracted from soln. of pH > 3 by thenoyltrifluoroacetone in benzene. The max. absorption of the yellow benzene layer is at 410 mμ, but the reagent also absorbs strongly in this region. At 430 mμ, reagent interference is negligible and Beer's law is followed (5 to 60 μg of U). The colour is stable for at least 72 hr.; citrate, tartrate, oxalate and CO_3^{2-} interfere.

P. D. Parr-Richard

2951. **Radiometric analysis of uranium content in ores.** P. E. Damon and H. W. Feely (Isotopes, Inc., Westwood, N.J.). *U.S. Atomic Energy Comm.*, Rep. RME-3153, July 31, 1957, 55 pp.—A radiometric method is presented for the rapid determination of the U content of ore. The method is valid regardless of the equilibrium state of the sample material. Uranium concn. is determined by direct measurement of the 0·093 MeV γ-ray of $^{234}Th(UX_1)$, the first daughter of ^{238}U. Resolution of this relatively low energy γ-ray, normally difficult because of the masking effects of the many other γ-rays present, is accomplished by combining three devices—copper absorbers to reduce, by absorption, the detection of γ-rays of energy < 0·093 MeV; thin (2 to 3 mm) NaI(Tl) scintillation crystal, to reduce the counting efficiency of higher energy γ-rays; critical absorption by lead of the 0·093 MeV γ-ray (critical absorption edge = 0·090 MeV). By using the copper absorbers and thin crystal, all γ-rays except those in the range of about 0·070 to 0·120 MeV are poorly detected. The very strong absorption by lead of the 0·093-MeV γ-ray of ^{234}Th may then be observed and related to the concn. of that isotope. Results indicate that a linear relationship exists between the U content and the experimentally determined ^{234}Th activity. The error of the method for most samples with a U content > 0·2% appears to be ≯ 20%. Nucl. Sci. Abstr.

2952. **Determination of carbon in uranium metal by combustion and absorption of carbon dioxide.** (U.K.A.E.A., Industrial Group, Chemical Services Dept., Springfields, Lancs., England). U.K.A.E.A. Report IGO-AM/S-48, 1959, 9 pp.—The sample (10 g) of uranium turnings that have been cleaned with HNO_3 (1 : 1) is burned, first in a mixture of N and O corresponding to air and then in O alone. The evolved CO_2 is dried and weighed after absorption in soda-asbestos. The coeff. of variation is ± 4% for contents of carbon between 600 and 700 p.p.m. G. J. Hunter

2953. **Infra-red spectra of uranyl nitrate hydrates and rubidium uranyl nitrate.** B. M. Gatehouse and A. E. Comyns (University College, London, W.C.1). *J. Chem. Soc.*, 1958, 3965–3971.—The i.r. spectra of uranyl nitrate di-, tri- and hexa-hydrate have been examined from 3700 to 700 cm^{-1} with N of normal isotopic composition and with N enriched with ^{15}N. The i.r. spectrum of rubidium uranyl nitrate is also reported. N. E.

2954. **Use of anion exchange in the spectrographic determination of impurities in plutonium.** R. Ko (General Electric Co., Hanford Atomic Products Operation, Richland, Washington). *U.S. Atomic Energy Comm.*, Rep. HW-57873, Oct. 20, 1958, 12 pp.—Impurity concn. of Al, Ca, Cr, Fe, Mg, Mn, Ni and Zn in plutonium were determined by an anion exchange-spectrographic method. Plutonium was separated by adsorption of the nitrate complex on Dowex-1 resin. The impurities were determined in the percolate by d.c. arc excitation, with Co as the internal standard. Concn. as low as 2 parts of impurity per 10^6 parts of plutonium in 50-mg samples were determined with a precision of ± 12% (95% confidence limits for a single measurement). Nucl. Sci. Abstr.

2955. **Separation of americium and promethium.** R. S. Pressly (Oak Ridge National Lab., Tenn.). *U.S. Atomic Energy Comm.*, Rep. ORNL-2202, Mar. 27, 1957, 4 pp.—Promethium-147 is separated with the long-lived rare-earth-element fraction in the process of obtaining ^{137}Cs from radioactive waste material. Because of the fission yield of 2·6% and the half-life of 2·6 years, there are contained in two-year decayed soln. approx. equal activities of ^{147}Pm and ^{137}Cs. Ion-exchange methods of separating the individual rare-earth elements separate Pm and Am together. These two activities are difficult to separate from each other. Ion-exchange methods separate small amounts effectively. With larger amounts, the pptn. method described in this report has been used and has proved to be more efficient than ion exchange. By this method Pm and Am are co-pptd. as fluorides. This fluoride ppt. is dissolved in H_3BO_3 and HNO_3 soln. The resulting soln. is made 3 *M* in H_2SiF_6, which, upon heating and slow digestion, decomposes into two volatile gases, HF and SiF_4. Promethium fluoride precipitates from the soln., and Am remains complexed in soln. Nucl. Sci. Abstr.

2956. **Amperometric titration of the fluoride ion.** J. J. den Herder and J. A. C. van Pinxteren (Rijksuniv., Utrecht). *Pharm. Weekbl.*, 1958, **93**

(**23**), 1013–1023.—Fluorides can be titrated amperometrically with $FeCl_3$. A rotating platinum electrode is used with a S.C.E. as a reference. The sample should contain between 38 and 950 μg of fluoride in 5 to 10 ml of 50% aq. ethanol saturated with NaCl in the pH range 4·5 to 5·5. During the titration, the vessel is cooled in ice. Interference is caused by CO_3^{2-}, HCO_3^- and PO_4^{3-}, but not by SO_4^{2-}. The titration can be repeated to $\pm$ 0·01 ml of titrant. For the determination of fluoride in drinking water, the fluorides are concentrated by evaporation, steam-distillation in the presence of H_2SO_4 and further evaporation.

G. BURGER

2957. **Separation of fluoride ions from interfering anions and cations by anion-exchange chromatography.** A. C. D. Newman (Rothamsted Expt. Stn., Harpenden, Herts., England). *Anal. Chim. Acta*, 1958, **19** (5), 471–476.—Fluoride ions in calcium phosphate are determined volumetrically after isolation by means of an anion-exchange resin. The fluoride must be sol. in alkaline EDTA (disodium salt) (**I**). *Procedure*—Digest the ground sample ($\not>$ 0·2 g) with 6 ml of *N* HCl for 24 hr. Add 3 ml of alkaline (**I**) soln. [93 g of (**I**) and 60 ml of aq. NH_3 (0·880) per litre], heat to 70° for a few min., cool and filter. Wash three times with H_2O and dilute the filtrate and washings to 250 ml. Pass 10 ml through a column of De-Acidite FF (Cl) and wash with 10 ml of H_2O. Treat the column with eluent soln. (53·5 g of NH_4Cl per litre with aq. NH_3 to pH 9·2, diluted 1:40). Reject the first 15 ml of eluate and collect the next 100 ml. Titrate 10 ml with chloroacetic acid (**II**) (0·1 *M*) to bromothymol blue (*a* ml). Dilute 40 ml of eluate plus **II** equiv. to 8*a* ml to 50 ml and titrate an aliquot (15 ml) with $Th(NO_3)_4$ soln. (5×10^{-4} *M*) with alizarin red S as indicator and photometric determination of the end-point.

T. R. ANDREW

2958. **Polarographic determination of chlorate.** L. Meites and H. Hofsass (Polytechnic Inst. of Brooklyn, N.Y., U.S.A.). *Anal. Chem.*, 1959, **31** (1), 119–122.—The method described is based on the rapid stoicheiometric reaction between chlorate and excess of Fe^{2+} in 3 *N* HCl. Chlorate soln. as dilute as 10^{-6} *M* can be determined with an accuracy of about $\pm$ 1%. Perchlorate and chloride do not interfere.

K. A. PROCTOR

2959. **Rapid spectrophotometric determination of manganese - triethanolamine and peroxide complexes of manganese(III).** E. R. Nightingale, jun. (Ethyl Corp., Detroit 20, Mich., U.S.A.). *Anal. Chem.*, 1959, **31** (1), 146–148.—Bivalent Mn is air-oxidised in the presence of alkaline triethanolamine to form a green complex with max. absorption at 438 mμ; Cr^{III} interferes seriously and Co^{IV} and Ni^{II} interfere slightly. The accuracy of the method is about $\pm$ 0·5%.

K. A. PROCTOR

2960. **The manganese content of sodium chloride.** Teru Yuasa. *Rep. Tokyo Chem. Ind. Res. Inst.*, 1958, **53** (10), 360–364.—Magnesium is used as a collector of Mn during its isolation from conc. soln. of NaCl. Chloride is first removed by evaporation with H_2SO_4, then Mn is determined colorimetrically by oxidation to permanganate in acid soln.

I. JONES

2961. **Chemical analysis of manganese ores.** S. Král (SONP, Kladno, Czechoslovakia). *Hutn. Listy*, 1958, **13** (10), 925–927.—The determination of the following constituents of manganese ores is discussed—total Mn ($\simeq$ 50%) (titration with $KMnO_4$); MnO_2 ($\simeq$ 60%) (indirect determination with an excess of $FeSO_4$ and back-titration with $KMnO_4$); SiO_2 (1 to 6%) (gravimetrically); Fe_2O_3 (3 to 10%) (titration with $KMnO_4$ after reduction with $SnCl_2$); TiO_2 (0·03 to 0·2%) (colorimetry with H_2O_2); CaO (0·03 to 0·15%) (titration with $KMnO_4$ after separation as oxalate); MgO (0·30 to 0·80%) (gravimetrically as $Mg_2P_2O_7$); total S (gravimetrically as $BaSO_4$); S^{2-} (0·50 to 0·70%) (combustion to SO_2 and iodimetric determination); P_2O_5 (0·07 to 0·10%) (gravimetrically as ammonium molybdophosphate); Al_2O_3 (2 to 3%) (gravimetrically); NiO (separation with dimethylglyoxime and photometric determination, or potentiometric titration with KCN); CuO (0·05 to 0·10%) (potentiometric determination with KCN, or photometry with aq. NH_3); SnO_2 (0·01 to 0·02%) (polarography in HCl and NH_4Cl after addition of hydrazine sulphate); Co_3O_4 (0·03 to 0·15%) (gravimetrically with 1-nitroso-2-naphthol); PbO (0·02 to 0·04%) (gravimetrically as $PbCrO_4$); ZnO (0·30 to 0·50%) (polarographically as ZnO_2^{2-}). Carbon dioxide and H_2O were determined by the usual methods.

J. ZÝKA

2962. **Analytical applications of the complexones. XII. Application of 1:2-(2-aminoethoxy)ethane-NN′-tetra-acetic acid to qualitative analysis. Detection of iron and vanadium.** F. Bermejo Martínez and M. Paz Castro (Fac. de Ciencas, Santiago de Compostela, Spain). *Inf. Quím. Anal.*, 1958, **12** (6), 157–159.—To the acid test soln. are added a few drops of a 5% soln. of the sodium salt of the reagent; a yellow colour is produced in the presence of Fe^{3+}. The colour may appear green in the presence of Cu^{2+}. Sufficient KHF_2 is added to complex the Fe and any Ti present, followed by a few drops of 30% H_2O_2 soln.; a yellowish-orange colour is produced in the presence of V^{5+}. The sensitivity of the method is 0·18 μg of Fe per ml and 0·61 μg of V per ml.

G. H. FOXLEY

2963. **Cerimetric titration of iron using a mixed indicator.** W. R. Heumann and C. Allard (Dept. of Chem., Univ. of Montreal, Canada). *Anal. Chem.*, 1959, **31** (1), 155–156.—In the procedure described previously (*cf. Anal. Abstr.*, 1958, **5**, 514), if Na diphenylbenzidinedecasulphonate (**I**) is used as warning indicator the error is only one-quarter of that caused by using Na diphenylaminesulphonate for obtaining the same intensity of colour. The total indicator-error ($\simeq$ 0·08%) is only about one-third. The recommended amount of **I** is 8 to 10 drops of 0·001 *M* soln. in a titration requiring $\simeq$ 40 ml of 0·1 *N* ceric soln.

W. J. BAKER

2964. **A new method for the titrimetric determination of the ferricyanide ion.** F. Lucena-Conde and I. Sánchez Bellido (Anal. Chem. Lab., Univ., Salamanca, Spain). *Talanta*, 1958, **1** (4), 305–309.—Conditions for the potentiometric titration of $Fe(CN)_6^{3-}$ with mercurous perchlorate in the presence of KSCN have been studied. The pH should be between 3 and 7, the SCN^- should be in a concn. of at least molar, and added to the $Fe(CN)_6^{3-}$ just before the titration, which should be carried out at $\not>$ 20°. The following procedure is recommended. Treat the soln. [0·4 to 3·0 milliequiv. of $Fe(CN)_6^{3-}$] with 5 ml of acetic acid (5 *N*),

dilute to 100 ml, add a further 5 ml of acetic acid and 20 ml of KSCN soln. (5 *N*). Immediately titrate at a temperature of $< 20°$ with 0·1*N* mercurous perchlorate (Pugh, *J. Chem. Soc.*, **1937**, 1824; **1945**, 588), detecting the end-point by means of a pH meter with platinum or glass indicator electrode and calomel reference electrode. Under these conditions the coeff. of variation of the method is $\pm$ 0·26%. R. E. ESSERY

2965. **Identification and colorimetric determination of ferricyanide ion with *o*-dianisidine.** F. Buscaróns and J. Artigas (Anal. Chem. Dept., Univ., Barcelona, Spain). *Anal. Chim. Acta*, **1958**, **19** (5), 434–437.—Within the pH range 1 to 2·8, *o*-dianisidine (**I**) gives an intense red colour with $Fe(CN)_6^{3-}$. Max. absorption is at 470 mμ, at which wavelength an extinction of 0·15 is recorded for 1 p.p.m. of $Fe(CN)_6^{3-}$ in a 1-cm cell. Other oxidising agents interfere. *Procedure*—Transfer the soln. to a 25-ml flask and add 2 ml of H_2SO_4 (2 *N*), 2 ml of $ZnSO_4$ soln. (2 *N*) and 2 ml of **I** (0·5% in 50% v/v acetone). Dilute to 25 ml and record the extinction 5 to 25 min. after addition of the reagent. T. R. ANDREW

2966. **Phase analysis of nitrided steel.** N. I. Blok, M. N. Kozlova, N. F. Lashko and A. G. Andreeva. *Zavod. Lab.*, 1958, **24** (11), 1315–1319.—Anodic dissolution is carried out in a mixture of 50 ml of conc. HCl and 1150 ml of methanol at $-7°$ with a c.d. of 0·02 to 0·025 amp. per sq. cm for 20 to 30 min. G. S. SMITH

2967. **Mass-spectrometric determination of gases in cast iron.** E. Kato. *Waseda Univ. Rep. Castings Res. Lab.*, 1957, (8), 81–84.—A description is given of the construction of a vacuum furnace connected to a mass spectrometer, and its use for determining the gas content in cast iron. In determining N, it was found that most of the N was extracted at 1350°, complete removal being obtained at 1650°. Results compared well with chemical values.
ABSTR. J. IRON ST. INST.

2968. **Determination of hydrogen in steel by a carrier-gas technique.** C. E. A. Shanahan and F. Cooke (Central Res. Lab., Richard Thomas & Baldwins, Ltd., Whitchurch, Aylesbury, Bucks., England). *J. Iron St. Inst.*, 1958, **190** (4), 381–385.—A diagram of apparatus and a detailed description are given of a method in which the sample is heated at 650° in a stream of N. The liberated H is converted, by passage over copper oxide - ferric oxide, into H_2O, which is determined by the Karl Fischer method. The accuracy is comparable with that of vacuum-heating methods.
J. W. O. PYEMONT

2969. **Improved vacuum-fusion method for the determination of oxygen and nitrogen in metals.** N. A. Gokcen. *Trans. Met. Soc. A.I.M.M.E.*, 1958, **212**, 93–100.—The construction and operation of a simple and accurate vacuum-fusion apparatus is described. Repeated analyses of Bureau of Standards samples show that they are inadequate for the acceptable range of O. The limits of interference of Mn, Al and Ti are established and the effects of Sn and water-cooling of the furnace tube are discussed.
ABSTR. J. IRON ST. INST.

2970. **Direct determination of combined carbon in grey pig-iron and cast iron.** H. Krapp and K.-H. Tytko. *Giesserei*, 1958, **45**, 639–642.—Combined C is determined directly by burning the hydrocarbons evolved on heating with H_2SO_4 in a stream of O on a glowing platinum spiral, and determining the resulting CO_2 volumetrically. The error is less than that obtained by difference using routine methods. ABSTR. J. IRON ST. INST.

2971. **Quantitative spectrographic determination of carbon in steel.** A. Pokorný (Závody J. Švermy, Brno, Czechoslovakia). *Hutn. Listy*, 1958, **13** (10), 892–899.—A Q24 quartz spectrograph, an FF20 spark generator and spectral lines C III 2296·86 A and Fe 2308·77 A, 2304·73 A, 2294·41 A, 2292·52 A, 2295·87 A, 2286·15 A, 2296·71 A and 2279·92 A have been used for the rapid determination of carbon in steel. Only one standard electrode is used and the content of C is directly read by means of a graduated ruler (illustrated). The procedure permits the analysis of steel containing 0·05 to 1·94% of C with an error of $\pm$ 3·1%. J. ZÝKA

2972. **Determination of magnesium in nodular graphite cast iron.** R. C. Rooney and B. Carter (B.C.I.R.A., Alvechurch, Birmingham, England). *J. Res. Brit. Cast Iron Ass.*, 1958, **7** (9), 442–445.—The rapid and accurate solvent extraction method described consists in the removal of Fe by extraction with *iso*butyl acetate, and of other interfering elements (*e.g.*, Ni, Mn and Al) as their diethyldithiocarbamates and cupferron complexes by extraction with $CHCl_3$, followed by titration of Mg with EDTA soln., with Eriochrome black T as indicator, in the presence of aq. NH_3. The results compared favourably with those of determinations made by the spectrographic method and by Green's method (*cf. Anal. Abstr.*, 1956, **3**, 353).
C. H. COWPER-COLES

2973. **Determination of small amounts of aluminium in cast iron.** R. C. Rooney (B.C.I.R.A., Alvechurch, Birmingham, England). *J. Res. Brit. Cast Iron Ass.*, 1958, **7** (9), 436–441.—A spectrophotometric method is developed for the rapid and accurate determination of down to 0·001% of Al in cast iron. The procedure comprises removal of Fe by *iso*butyl acetate and other interfering elements (*e.g.*, Mn) by Na diethyldithiocarbamate, extraction of Al with cupferron and $CHCl_3$, conversion of the Al into its 8-hydroxyquinoline complex and measurement of the extinction at 395 mμ, with a 1-cm cell and a Uvispek spectrophotometer. The method is applicable to steel, and total Al can also be determined. C. H. COWPER-COLES

2974. **Determination of aluminium in iron and steel.** Chin-Hwei Jen and Chung-Hsian Tao. *Chem. World*, 1958, **13** (5), 210–213.—Sajo's method for the determination of large amounts ($> 1\%$) of Al present in iron and steel (the indirect titration of Al with EDTA) is discussed. Dissolve the sample in aqua regia, evaporate to dryness and extract the residue with conc. HCl (10 ml) to remove W. Dilute to 200 ml, reduce Fe^{3+} with 3 g of Na_2SO_3 and separate Al, Cr, etc., from other metals by the ammonium benzoate method. Dissolve the benzoates in HCl (1:3) (30 ml) and at pH 4·5 to 5·0 and 40° to 45° back-titrate any excess of EDTA with standard Zn^{2+} soln., with $Fe(CN)_6^{3-}$ - $Fe(CN)_6^{4-}$ and benzidine soln. (freshly prepared) as indicator. Add excess of NaF to liberate the EDTA combined with Al and titrate as before. If the ratio of Cr to Al is $> 1:5$, the deep-purple colour of the complex formed by Cr^{3+} with EDTA will obscure the end-point. The procedure may be modified by dissolving

the benzoates in *N* NaOH (30 ml), oxidising Cr with H_2O_2 and finally separating Al^{3+} from CrO_4^{2-} by the ammonium benzoate method. This modified procedure may be used for analysing samples containing Cr, Ni, Cu, W, Mo, V, Ti, etc. The time required for the simultaneous analysis of three samples is < 3 hr. SCI. ABSTR. CHINA

2975. **Polarographic determination of aluminium in steel and non-ferrous alloys after chromatographic separation.** M. Kroupa. *Hutn. Listy*, 1958, **13** (10), 922–925.—After the decomposition of the sample, Al, Ni, Ti and V and small amounts of Cr are retained on a cellulose column, while Fe, Cu, Mo and some other elements pass through. The zone containing Al, Ni, Ti, V and Cr is eluted with dil. HCl; Ti and V, and small amounts of Zr, Nb and Ta, are separated from Al by pptg. with cupferron and extracting with $CHCl_3$; Ni and Cr are separated by means of rapid electrolysis with a mercury cathode. There is no interference from Zn, Mg, Mn, Be, Ca, As or P. Aluminium forms a complex with Diamond Chrome Red A, the polarographic wave of which has been used for the method of determination described. The method is very sensitive; 1 μg of Al in 10 ml of soln. can be determined. The procedure has been tested by analysing various types of steel and alloys containing 0·001 to 1% of Al. Detailed procedures are given. J. ZÝKA

2976. **Determination of vanadium in high-alloy steels by isotope dilution.** G. Leliaert, J. Hoste and J. Eeckhaut (Lab. of Anal. Chem., Ghent Univ., Belgium). *Talanta*, 1958, **1** (4), 369–373.—Vanadium is titrated amperometrically, after removal of Cr as CrO_2Cl_2, losses of V by adsorption on insol. oxides being corrected for radiometrically. Dissolve the sample (0·1 to 1 g) in HCl, add HNO_3, evaporate and fume with $HClO_4$. Transfer to a platinum dish with HF, add a suitable amount ($\simeq$ 500,000 counts per min.) of carrier-free ^{48}V and heat. The oxides are thus dissolved, and are pptd. again on heating to fumes, and are removed by slight dilution and filtration through asbestos. Again fume, and pass dry HCl gas through the boiling mixture for 10 min. to eliminate Cr. Boil to remove HCl, then dilute to 50 ml, add a few drops of 1% $FeSO_4$ soln. in 2% H_2SO_4 to reduce the V to V^{IV}, then add 0·05 *N* $KMnO_4$ to give a faint pink, the excess being destroyed, after 5 min., by adding 2 to 3 drops of 2% $NaNO_2$ soln. followed by 0·5 g of urea. Then titrate the V amperometrically with standard $FeSO_4$ soln. (0·0025 to 0·025 *N* according to the V content of the sample), with a rotating platinum electrode. A polarising voltage of 0·85 V *vs.* the S.C.E. was found satisfactory for soln. containing 4 to 40 micro-equiv. of V per 50 ml. After titration, evaporate the soln., make up to 50 ml, and count 4 ml in a well-type detector. The method of calculation of the result, which includes the correction for adsorption losses, is given. The accuracy is better than 1% and results for six U.S. Nat. Bureau of Standards samples agree well with the N.B.S. results.
R. E. ESSERY

2977. **Rapid spectrographic determination of niobium and silicon in steel.** O. Bělohlávek (SONP Kladno, Czechoslovakia). *Hutn. Listy*, 1958, **13** (10), 920–922.—A Q24 spectrograph, an FF20 spark generator and spectral lines 3130·786 A (Nb), 3083·742 A and 2518·101 (Fe) and 2516·123 A (Si), have been used, together with other modifications, for the determination of Nb and Si in various types of steel. The method is suitable for a rapid routine analysis (35 to 40 min. for one sample, 2 hr. for 10 samples) with an accuracy of 7 to 8%. The determination of Nb in the presence of Ta and the determination of both elements are discussed. The method has been tested on steel containing 0·40 to 1·40% of Nb and 0·20 to 1·10% of Si. J. ZÝKA

2978. **Simultaneous potentiometric determination of molybdenum and tungsten in special steels.** R. Suarez Acosta (Inst. del Hierro y del Acero, Madrid, Spain). *Inst. Hierro y Acero*, 1958, **11**, 282–286.—The sample soln. is reduced with zinc and conc. HCl followed by 0·1 *N* $CrSO_4$, and titrated potentiometrically with 0·04 *N* $K_2Cr_2O_7$ in an atmosphere of N. Breaks in the titration curve correspond to the oxidation of excess of Cr^{2+}, Mo^{5+} and W, respectively. The method is rapid and accurate. G. H. FOXLEY

2979. **Methods for the analysis of iron and steel. Part 40: Tungsten in iron and steel (gravimetric method).** British Standards Institution (2 Park St., London). B.S. 1121:Part 40:1959, 8 pp.—The procedure described is suitable for steels containing no Ta or Nb and having a W content exceeding 3%. After solution of the sample (2 g) in HCl, W is pptd. as the hydrated oxide. Cinchonine is added to complete the separation and the crude oxide is ignited in platinum at 800°. Silica is removed by evaporating to dryness with HF, and the oxide residue is then weighed, fused with Na_2CO_3 and the melt extracted with water. Insoluble matter is filtered off, ignited and weighed. The filtrate is examined for Cr, V and Mo, and the weight of the impure tungstic oxide found is corrected for the presence of these elements and the insoluble matter. A precision of $\pm$ 0·04% is claimed at the 3% W level and $\pm$ 0·15% at the 18% W level. T. R. ANDREW

2980. **Activation analysis of tungsten in high-alloy steels.** G. Leliaert, J. Hoste and J. Eeckhaut (Lab. of Anal. Chem., Univ. of Ghent, Belgium). *Talanta*, 1959, **2** (2), 115–123.—The method for V (*cf. Anal. Chim. Acta*, 1958, **19**, 100) is applied to the determination of W, the sample being exposed to a high neutron flux and the decay of activity measured. The properties of the most important isotopes produced by steel components are discussed, and two methods are described. In the direct method, when precautions are taken against self-shielding, the activity due to ^{187}W can be computed by a graphical analysis of the decay curve, provided that the ratios of Cu, As and Mo to W do not exceed 0·2, 0·01 and 5, respectively, but the procedure takes 15 days. For the separation of W, the irradiated sample is dissolved in HCl, and treated with HNO_3, $HClO_4$ and H_2O_2 by a specified procedure, after which Na_2WO_4 and Na_2VO_4 are added and the mixture is heated to precipitate tungstic acid. This is filtered off and washed with 3% HNO_3 and the ppt. and paper are counted together or separately. The recovery of W is better than 99·8%, and satisfactory radiochemical separation from most of the steel components is achieved. Niobium and Ti do not interfere, but Ta is co-pptd. with W, and an analysis of the decay curve is then required. The accuracy and precision are similar to those of a careful chemical analysis. R. E. ESSERY

2981. **Determination of cobalt in iron and steel.** R. C. Rooney (British Cast Iron Res. Ass., Alvechurch, Birmingham). *Metallurgia, Manchr*, 1958, **58**, 205–208.—After extraction of Fe and Cu with cupferron and $CHCl_3$, Co is determined photometrically as the 2-nitroso-1-naphthol complex in benzene. For Co contents in the range 0·001 to 3%, the sample (1 g) is dissolved in the cold in HCl (25 ml) and HNO_3 (5 ml), and diluted to 100 ml. A 10-ml aliquot is taken (for 0·3 to 3% of Co, 5 ml is taken from an initial 500 ml) and 25 ml of aq. cupferron soln. (6%) is added, the soln. is shaken, $CHCl_3$ (30 ml) is added, and the mixture is shaken for 30 sec. The aq. phase is separated and washed with 10-ml portions of $CHCl_3$ until the $CHCl_3$ extracts are colourless. The aq. layer is then evaporated to dryness and organic matter destroyed by fuming with HNO_3 (5 ml) and $HClO_4$ (sp. gr. 1·54, 3 ml). The cooled soln. is diluted to $\simeq$ 25 ml and transferred to a separating-funnel. Sodium acetate soln. (25 ml, 2 *M*) and methanolic 2-nitroso-1-naphthol soln. (10 ml of 0·1% for every 3 mg of Co plus Ni present) are added. After 2 min. the mixture is extracted with 20 ml of benzene and the extinction of the extract is measured against benzene, for 0·5 to 15 μg of Co at 365 mμ, and for 2 to 120 μg of Co at 539 mμ. For Co contents in the range 0·00001 to 0·006%, a 5-g sample is taken, and a preliminary separation of $FeCl_3$ by extraction with *iso*butyl acetate is made. The aq. phase is evaporated to dryness, organic matter is destroyed with HNO_3 and $HClO_4$, and the extraction with cupferron, etc., is carried out as described above. T. R. ANDREW

2982. **Qualitative detection and colorimetric determination of cobalt by means of N-oximinoacetylanthranilic acid.** F. Buscaróns and L. M. Munné (Lab. of Anal. Chem., Univ., Barcelona, Spain). *Anal. Chim. Acta*, 1958, **19** (5), 432–434 (in French).—This reagent reacts with Co at pH 8·5 to 9·5 to give a wine-red colour; Ni gives a similar colour which may be destroyed by the addition of KCN; Cu^{2+} and Fe^{3+} give green and brown colours, of low sensitivity. The optimum wavelength for photometric determination is 430 mμ.

T. R. ANDREW

2983. **Titrimetric determination of small amounts of cobalt with an equivalent ratio of 1:37.** L. G. Bartha and S. Görög (Inst. for Inorg. and Anal. Chem., Univ. of Szeged, Hungary). *Talanta*, 1958, **1** (4), 310–313.—The Co is pptd. as $KPb[Co(NO_2)_6]$ in the presence of a little K_2SO_4, under specified conditions. The washed ppt. is boiled under specified conditions with NaOH, liberating NO_2^-, which are then reduced with boiling standard $FeSO_4$ soln. On the macro-scale (0·3 to 3 mg of Co), the excess of Fe^{2+} is back-titrated with 0·1 *N* $KMnO_4$. The difference between this titration and a blank, multiplied by 0·1593, gives Co (mg). On the micro-scale (30 to 300 μg of Co), the mixture after boiling with $FeSO_4$ soln. is treated with HCl and a few marble chips, and cooled to 40° to 50°. Potassium iodide (5 g) is added, and the iodine liberated by the Fe^{3+} formed in the reaction is titrated with 0·01 *N* $Na_2S_2O_3$. After correction for a blank, 1 ml $\equiv$ 15·93 μg of Co. The error of the macro-procedure is $\simeq \pm 1\%$ and that of the micro-procedure $\simeq \pm 5\%$, but the latter increases with decreasing amounts of Co below 40 μg.

R. E. ESSERY

2984. **Analytical applications of the complexones. XIII. Absorptiometric and spectrophotometric micro-determination of cobalt with 1:2-diaminocyclohexanetetra-acetic acid.** F. Bermejo Martínez and R. Rey Mendoza (Fac. de Ciencas, Santiago de Compostela, Spain). *Inf. Quim. Anal.*, 1958, **12** (6), 160–164.—The test soln. containing Co^{2+} is treated with a 5% soln. of the disodium salt of the reagent (2 ml) and 30% H_2O_2 soln. (5 ml), followed by 2·5% aq. NH_3 (2 ml). After being heated for 5 min., the soln. is cooled, diluted to 25 ml and its extinction measured at 542 mμ, with an Ilford filter No. 604. The absorption of the complex is a linear function of the Co concn. between 2 and 125 μg per ml. There is no interference from Fe^{3+}, Mn^{3+}, Cu^{2+}, Ni^{2+} or Cr^{3+}. G. H. FOXLEY

2985. **Colorimetric determination of a trace of cobalt in nickel with anion-exchange resin.** Atsushi Mizuike, Yoshio Iida and Shizo Hirano (Inst. Techno-anal. Chem., Fac. of Engng, Tokyo Univ., Hongo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1958, **61** (11), 1459–1460.—Separation of Co from Ni by Kraus's method (*J. Amer. Chem. Soc.*, 1952, **74**, 843) with Amberlite IRA-400 (Cl) (60 to 100 mesh) was examined with radioactive ^{60}Co and found to be applicable to the analysis of Mond nickel (Co $\simeq$ 8 p.p.m.). The Co on the resin column (2 cm × 5 cm) is eluted with 0·1 *N* HCl and determined colorimetrically with nitroso-R salt. K. SAITO

2986. **Magnetic analysis by electron diffraction of the nickel occluded in nickel oxide.** S. Yamaguchi (Inst. Phys. and Chem. Res., Tokyo, Japan). *Z. anal. Chem.*, 1959, **165** (2), 105–109.—The Lorentz effect in electron diffraction is utilised for the magnetic analysis of Ni occluded in NiO. A powder consisting of Ni and NiO, and a Ni - NiO film serve as test samples. Measurement of the spontaneous magnetism of the sample allows quant. determination of Ni in its oxide. The test is illustrated with photographs of the effect.

G. P. COOK

2987. **Electrochemical phase analysis of alloys.** Yu. A. Klyachko, M. M. Shapiro, V. S. Mal'tseva and V. A. Mil'chev (Central Sci. Res. Inst. of Ferrous Metall.). *Zavod. Lab.*, 1958, **24** (11), 1308–1314.—Phase analysis of nickel-based alloys (nimonic) requiring the separation of at least three phases is studied. Some general conclusions concerning the theory of the anodic dissolution method for phase analysis of alloys are formulated.

G. S. SMITH

2988. **Polarographic determination of ruthenium.** M. V. Šušić. *Bull. Inst. Nucl. Sci. "Boris Kidrich", Belgrade*, 1958, **8**, 53–57 (in English).—A polarisation curve was obtained for a mixture of Ru^{3+} and Ru^{4+} with a dropping-mercury electrode at different pH values in 0·9 *M* $HClO_4$, 0·1 *M* Complexone II (EDTA) (**I**), and a mixture of 0·05 *M* **I** and ammonium acetate as supporting electrolytes. The polarisation curve consisted of three separate waves corresponding to catalytic waves of hydrogen, the height of which depended on the pH value. The change of $E_{\frac{1}{2}}$ as a function of the pH was investigated. For some pH values $i_d/C = k$ and therefore ruthenium could be determined at very low concn. The third wave was used for the polarographic determination of very low concn. of ruthenium ($< 10^{-7}$ g per ml) in 0·9 *M* $HClO_4$. The first wave was used for concn. $> 1 \times 10^{-7}$ g per ml.

K. R. C.

2989. **Radiochemical determination of fission ruthenium in aqueous solutions. A non-distillation technique.** R. R. Rickard and E. I. Wyatt (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1959, **31** (1), 50–51.—The procedure described depends on the use of KIO_4 and sodium hypochlorite to retain Ru as ruthenate and per-ruthenate in an alkaline soln., whilst the other fission products are removed by co-pptn. on zirconium hydroxide. The Ru is finally isolated as the metal for counting; decontamination factors of $\simeq 10^4$ can be attained. The precision is $\pm 4\%$ and a duplicate analysis can be made in $\simeq 1$ hr. Citrate, oxalate, phosphate and mineral acids do not interfere, but tartrates must be absent.

W. J. BAKER

2990. **2-Mercaptobenzimidazole as a reagent for palladium.** J. Xavier (Dept. of Inorg. Chem., Indian Ass. for the Cultivation of Sci., Calcutta). *Z. anal. Chem.*, 1958, **164** (2), 250–254 (in English).—*Procedure*—Up to 0·25 mg of Pd in dil. HCl soln. (15 to 20 ml) is taken for test. Adjust the pH to 1·0 to 2·6, add 1 ml of 2-mercaptobenzimidazole soln. (1% w/v in 95% ethanol) and make up to 25 ml with H_2O. After 10 min. measure the extinction at 380 mμ *vs.* a reagent blank. Small amounts of Fe^{3+} are masked with 2 to 5 ml of 10% H_3PO_4 soln. For 2 p.p.m. of Pd, tolerance limits in p.p.m. for other ions are—Co^{2+} (400), Ni^{2+} (20), Cd^{2+} (600), Mn^{2+} (500), Cr^{3+} (50), Pb^{2+} (500), Cu^{2+} (20), Fe^{3+} (150 with H_3PO_4), Fe^{2+} (200) and molybdate (40). Common anions do not interfere. The sensitivity is 0·04 μg of Pd per ml.

P. D. FARR-RICHARD

2991. **3-Aminopicolinic acid as a reagent for the gravimetric and volumetric estimation of palladium.** A. K. Majumdar and S. P. Bag (Jadavpur Univ., Calcutta, India). *Z. anal. Chem.*, 1958, **164** (4), 394–398 (in English).—Milligram amounts of Pd can be determined gravimetrically by pptn. at 100° with aq. 3-aminopicolinic acid (1%, w/v) from *N* HCl soln. (preferably) or from soln. in the pH range 1 to 12·5. The complex, $Pd(C_6H_5O_2N_2)_2$, is dried at 110° and weighed. Few ions interfere; Cu should be masked with EDTA and the Pd then pptd. at a pH < 3; when Ag is present, the pptn. should be carried out at a pH < 7. For a volumetric determination of Pd (3 to 20 mg), the pptd. complex is dissolved by warming with ammoniacal KCN soln. ($\simeq 0{\cdot}04\ M$), the excess of which is then back-titrated with standard $AgNO_3$ soln., 5% (w/v), aq. KI soln. being used as indicator.

W. J. BAKER

2992. **Spectrophotometric determination of palladium. II. Bismuthiol II as an analytical reagent.** A. K. Majumdar and M. M. Chakrabartty (Jadavpur Univ., Calcutta, India). *Anal. Chim. Acta*, 1958, **19** (5), 482–487.—Bismuthiol II reacts with Pd in the pH range 3 to 10 to give a stable red colour or ppt. Acetone or preferably 2-methoxyethanol (10% v/v) prevents pptn. of salts at high concn. The recommended wavelength for measurement is 420 mμ. Tolerance limits are tabulated for 21 cations and 21 anions. The extinction for 4 μg of Pd^{IV} is 0·317 at 420 mμ in a 1-cm cell. Beer's law is obeyed from 0·4 to 8·0 μg per ml. Platinum, Au, Cu, Cr, Fe, Hg, Ag, Tl, U, VO_3^- and CN^- interfere.

T. R. ANDREW

2993. **Gravimetric determination of osmium with 1:2:3-benzotriazole.** R. F. Wilson and L. J. Baye (Dept. of Chem., Texas Southern Univ., Houston, U.S.A.). *Talanta*, 1958, **1** (4), 351–354.—Osmium is pptd. with 1:2:3-benzotriazole (**I**) at pH 4, to give the stoicheiometric compound $Os(OH)_3.(C_6H_4NHN_2)_3$, with a factor of 0·3178. Transfer 10 ml of test soln. to a flask containing 1 ml of ethanol. Add 25 ml of a 2% aq. soln. of **I**, and heat the mixture containing the red complex on a steam bath for 15 min. Adjust the pH roughly to 3 with acetic acid, digest for a further 15 min. to coagulate the ppt. and filter on a medium-porosity sintered-glass crucible. Wash the ppt. with hot water, dry at 110° for 1 hr. and weigh. Recoveries of from 1·51 to 22·62 mg of Os showed errors of from $-$ 0·07 to $+$ 0·05 mg. The method is applicable to Os after distillation as OsO_4 from HNO_3 soln. The conditions are not critical and the ppt. is stable between room temp. and 200°.

R. E. ESSERY

2994. **Rapid analysis of silicate rocks by means of a spectrophotometer.** G. Ledent (Serv. Géol. Congo Belge, Leopoldville). *Bull. Serv. Géol. Congo Belge*, 1956, **6** (5), 12 pp.—The various elements are determined as follows—Al gravimetrically (by difference); Ca and Mg, complexometrically with EDTA; Si, Fe, Ti, Mn and P, spectrophotometrically; Na and K, flame-photometrically; H_2O by loss on ignition. Particular emphasis is placed on the accurate adjustment of the pH of the soln.

R. A. HOWIE

2995. **Rapid analysis of silicate rocks and minerals.** J. P. Riley (Dept. of Oceanography, Univ., Liverpool, England). *Anal. Chim. Acta*, 1958, **19** (5), 413–428.—Methods are described for the determination of Si, Ti, Fe, Al, Mn, P, Ca, Mg, Na, K, H_2O, CO_2 and Fe^{II} in silicate rocks. Silicon is determined by the procedure of Mullin and Riley (*cf. Anal. Abstr.*, 1955, **2**, 1686), on a 0·1-g sample after fusion with NaOH in a silver crucible. Water and CO_2 are determined by the method of Riley (*Analyst*, 1958, **83**, 42). Ferrous iron is determined by titration with $K_2Cr_2O_7$ soln. after dissolution in a mixture of HF and H_2SO_4 under non-oxidising conditions. The remaining elements are determined on aliquot portions of a main soln. prepared by dissolving a sample (0·5 g) in HF and $HClO_4$, evaporating to fumes and diluting to 500 ml. Titanium is determined photometrically with H_2O_2; Fe with 2:2′-dipyridyl; Al by extraction with 8-hydroxyquinoline in $CHCl_3$; Mn by persulphate oxidation to permanganate; P by a molybdenum blue method. Sodium and K are determined by flame photometry after removal of interfering elements by passage through an Amberlite IRA-400 (citrate) anion-exchange column. After separation of interfering elements (except Mn) by extraction with 8-hydroxyquinoline in $CHCl_3$, Ca and Mg are determined volumetrically with EDTA (disodium salt). Detailed instructions are given for carrying out these procedures. Six or seven samples may be analysed in one week. Results quoted for granite GI and diabase WI are in good agreement with accepted values.

T. R. ANDREW

2996. **Matrix corrections in X-ray spectrographic trace-element analysis of rocks and minerals.** J. Hower (Montana State Univ., Missoula, U.S.A.). *Amer. Min.*, 1959, **44** (1–2), 19–32.—The three matrix effects which must be accounted for in X-ray spectrographic analysis are attenuation of the incoming beam, mutual excitation of the elements, and absorption of the emitted fluorescent radiation. These effects may be determined by

the mass absorption coefficient of the rock or mineral; the relative absorptions of the elements are virtually constant at all wavelengths. A complete matrix correction can be made for Ni and heavier elements by determining the absorption of a sample relative to Al_2O_3 at one wavelength. The relative absorption can be determined either by calculation, if the major constituent analysis is known, or by means of an internal standard; partial matrix corrections can be made for the lighter elements Co, Mn, Cr, V and Sc.

R. A. HOWIE

2997. **Industrial analysis of natural fluorites.** P. Povondra and J. Vébr (Manganové Závody, Chvaletice, Czechoslovakia). *Chem. Listy*, 1958, **52** (10), 2008–2009.—The decomposition of fluorites with a mixture of H_3BO_3 and $HClO_4$ enables fluorine to be readily separated in the form of a fluoroborate complex. *Procedure*—Dry the finely powdered sample of natural fluorite at 100°, and to 1 g add $HClO_4$ (1 : 3) (15 ml) saturated at 50° with H_3BO_3, heat and evaporate to dryness. Cool, add conc. HCl and boiling H_2O (80 ml) and set aside for 10 min. Filter off and ignite the silicic acid, evaporate several times to dryness with HF and determine SiO_2 by difference. Heat the residue with a few ml of HCl (1:4), dilute with boiling H_2O, filter through a paper filter, ignite the filter, convert any Ba in the ash to $BaSO_4$ and weigh. If high results are obtained, fuse the $BaSO_4$ with Na_2CO_3 and repeat the procedure. Combine the filtrates, transfer to a 250-ml flask, dilute to vol. with H_2O and measure an aliquot for the determination of Ca. If low concn. of Fe and Al are present, dilute slightly, add triethanolamine soln. (30%) (5 ml), make alkaline with NaOH and titrate Ca with EDTA (disodium salt) soln., with murexide as indicator. Larger amounts of Fe and Al must be removed by hydrolytic pptn. with hexamine. A correction for the content of $CaCO_3$ can be made by carrying out a volumetric determination of CO_2.

J. ZÝKA

2998. **Application of the X-ray diffractometer for bauxite analysis.** F. Rohner (Forschungsinst. der Aluminium-Ind.-A.-G., Neuhausen am Rheinfall, Switzerland). *Chimia*, 1958, **12** (10), 287–291.—The böhmite and diaspore contents of bauxite are obtained by measurement of the intensities of the X-ray diffraction lines at 6·15 A and 3·97 A, respectively. A calibration curve is obtained by the analysis of known böhmite - diaspore mixtures. The results show a coeff. of variation ranging from $< \pm 1\%$ for a 95% diaspore content to $\simeq \pm 10\%$ for a 5% diaspore content. The ease with which a bauxite sample can be decomposed is directly related to its böhmite content.

J. H. WATON

2999. **Quantitative flame-photometric determination of potassium, sodium, calcium and aluminium in kaolin.** F. Hegemann and W. Hert. *Ber. dtsch. keram. Ges.*, 1958, **35**, 258–263.—The sample is decomposed with HF plus oxalic acid to separate the SiO_2 and CaO, and Na and K in the filtrate are determined flame-photometrically. The interfering effect of Al is compensated by the addition of the corresponding amount of Al to the standard soln. of the alkali metals. This relatively large amount of Al also suppresses the interference by Fe and Mg. For the determination of the Al, *n*-butanol is added to the test soln. as a "flame activator" and the interference of the alkali metals is compensated by adding the corresponding amount to the standard soln. of Al. The Ca oxalate separated by the initial treatment of the sample with HF and oxalic acid is dissolved in HCl and measured flame-photometrically against a standard soln. containing the same amount of HCl.

J. A. SUGDEN

3000. **A rapid method for the determination of ash in coal.** L. L. Arsin (Inst. za Ugal, Belgrade). *Bull. Soc. Chim. Belgrade*, 1956, **21** (6–10), 457–462.—The sample of coal is first ground to 0·2 mm mesh, and pellets of $\simeq$ 1 g are made with a press. Volatile matter is removed by heating for 5 min. at 500° to 600°. There is no danger of decrepitation as the pores in the pellets permit complete removal of water from the interior. The samples are next heated to 800° $\pm$ 10° in a few minutes, and then incinerated for 45 to 50 min. The longer incineration did not change the amount of ash formed and it appears that in this further treatment enough air penetrates the coal sample to ensure complete combustion. The new method takes only 1 hr. and is particularly suitable for coals rich in argillaceous matter. For coals rich in carbonaceous mineral matter both the new and the standard method of ash determination were found to be rather inaccurate owing to the formation of secondary SO_2 compounds by interaction of organically bound S with alkali components of the ash. (*Cf. Anal. Abstr.*, 1959, **6**, 3003.)

A. GROCHOWSKI

3001. **Rapid analysis of coal ash, slag and boiler deposits.** K. Archer, D. Flint and J. Jordan (Brit. Coal Utilis. Res. Ass., Randalls Rd., Leatherhead, England). *Fuel, Lond.*, 1958, **37** (4), 421–443.—Solutions (*A* and *B*) are prepared by fusing the sample with NaOH and by HF - H_2SO_4 treatment. Aliquots of *A* are used for the absorptiometric determination of Si (molybdenum blue) and Al (alizarin red S), and of *B* for Fe and Ti (tiron), P (molybdovanadophosphate), and Mn (MnO_4^-). Calcium and Mg are titrated with EDTA after complexing Fe, Al and Ti with triethanolamine, and Na and K by flame photometry. Sulphur is determined gravimetrically after extraction from the ash with aq. Br - HCl. Full details of procedure are given. Agreement with results by classical methods is satisfactory. One operator can analyse ten samples for eleven constituents in a week.

A. R. PEARSON

3002. **Critical appreciation of computations of the melting-point of ash [of coal] from the chemical analysis.** L. L. Arsin and B. M. Marković (Inst. za Ugal, Belgrade). *Bull. Soc. Chim. Belgrade*, 1956, **21** (6–10), 443–455.—Factors for computation of the melting-point of coal ash from the chemical composition of the ash, developed from formulae by Shaeffer, Prost, Teuner, Nicholls and Selvig, have been extensively checked on several samples of Yugoslav coal. In all cases the factor *vs.* m.p. diagrams were non-linear and none of the formulae could be successfully applied. It appears that all these methods ignore the formation of additional chemical compounds in the viscous state from the large amount of inorganic matter present in the coal.

A. GROCHOWSKI

3003. **Effect of aeration of the combustion furnace on the ash content of coal.** L. L. Arsin (Inst. za Ugal, Belgrade). *Bull. Soc. Chim. Belgrade*, 1956, **21** (6–10), 463–469.—Experiments were carried out with an oven adapted for better

aeration during the incineration of the coal samples for the ash analysis. A Lindberg electric combustion furnace, normally with one aperture (1 cm × 2 cm) in the centre of the door, had 11 apertures (1 cm) bored at the base of the door and two slits (2·2 cm) in the wall opposite. While aeration was of little importance for coals rich in argillaceous matter, for coals rich in carbonaceous matter the organically bound S tended to react with the alkali components of the ash and the final amount of ash was increased by secondary SO_3 compounds. The improved aeration of the oven permitted faster and more complete removal of volatile matter and therefore considerably reduced the formation of these compounds. A. GROCHOWSKI

3004. **Determination of several valencies of iron, arsenic, antimony and selenium in glass.** P. Close, H. M. Shepherd and C. H. Drummond (Owens-Illinois Tech. Center, Toledo, Ohio, U.S.A.). *J. Amer. Ceram. Soc.*, 1958, **41**, 455–460.—*Procedure for ferrous iron*—The sample is decomposed in the cold with HF, bubbling with CO_2 to expel SiF_4 and sulphides (from amber glass). After complexing the excess of HF with H_3BO_3, the soln. is titrated with $Ce(SO_4)_2$, with Fe^{II} - 1:10-phenanthroline as indicator. An accuracy of ± 0·01% is claimed. *Procedure for* (*a*) *tervalent arsenic and antimony*—The sample is decomposed with HF plus H_2SO_4 (or $HClO_4$), excess of HF is complexed with H_3BO_3 and the soln. is titrated with $Ce(SO_4)_2$, with OsO_4 as the oxidation catalyst. (*b*) *Total arsenic and antimony*—The sample is decomposed as in (*a*) with the addition of HNO_3; the As^V is reduced with hydrazine, the As^{III} is distilled off and the Sb^V is recovered as sulphide. (*c*) *Quinquevalent arsenic*—The sample is decomposed as in (*a*) the As^{III} is distilled off and the As^V determined as in (*b*). (*d*) *Total selenium*—The sample is decomposed with HF, HBr and Br and the excess of Br removed by heating with formic acid; excess of $Na_2S_2O_3$ is added and back-titrated with iodine (the end-point being determined potentiometrically with a platinum - tungsten electrode pair). (*e*) *Quadrivalent selenium*—The sample is decomposed with HF alone and after digestion with acetic acid and Ba acetate the filtered soln. is treated as in (*d*). (*f*) *Quadrivalent and sexavalent selenium*—The sample is decomposed with HF and, after filtering off the residue containing elementary Se the soln. is treated as in (*d*). (*g*) *Elementary selenium*—The residue from (*f*) is washed, and treated as in (*d*). J. A. SUGDEN

3005. **Successive determination of manganese, sodium and potassium in cement by flame photometry.** C. L. Ford (Portland Cement Assn., Chicago, Ill., U.S.A.). *Bull. A.S.T.M.*, 1958, (233), 57–63.—A procedure is given for determining Mn, K and Na in a soln. of cement (1 g) in dil. HCl with only one set of standard solutions containing known amounts of Na and K and Mn, plus Ca equiv. to 6300 p.p.m. of CaO. A Beckman spectrophotometer with flame- and photomultiplier-attachment is used or, for Na and K only, a Perkin - Elmer flame photometer. The wavelengths (mμ) used are 403·3 (Mn), 589 (Na) and 768 (K). No separations other than that of insol. matter are required. Results are within + 0·01% of those obtained by standard methods. W. J. BAKER

See also Abstracts—**2825**, Ferrous and non-ferrous metallurgy (review). **2829**, Reactions of phenylfluorone deriv. with metal ions. **2830**, Rubeanic acid deriv. as colorimetric reagents. **2831**, Reactions of some oxine derivatives with certain metals. **2833**, Determination of Zn, Co, Mn and Cu with ammonium N-2-naphthylmalonamate. **2842**, Spectrographic determination of O in metals and of trace elements in minerals. **3025**, Determination of ammonium compounds. **3090**, Detection of yellow P and phosphides in biological material. **3236**, Detection of Hg in air. **3248**, Determination of ^{90}Sr in sea water. **3249**, Separation of ^{90}Sr, ^{137}Cs, ^{144}Ce and ^{147}Pm from sea water. **3250**, Determination of ^{106}Ru in effluent and sea water. **3283**, Chromatographic separation of inorganic ions. **3316**, Analysis of steel and aluminium alloys. **3340**, Chloride ion analyser. **3356**, Analysis of mixtures of Fe, Mo and W.

3.—ORGANIC ANALYSIS

Determination of elements and radicals and of organic compounds not included in other sections. Organic industrial products, including petroleum and its products, fuels, detergents, volatile oils, cosmetics, dyestuffs, fibres, plastics, resins, paints, elastomers, leather, explosives.

3006. **Micro-determination of active hydrogen in organic compounds with aluminium lithium hydride.** K. N. Arjungi, R. S. Kulkarni and T. S. Gore (Dept. Chem. Technol., Univ. of Bombay, India). *J. Sci. Ind. Res., India, B*, 1958, **17** (11), 459–461.—A modified version of Roth's apparatus (*Mikrochemie*, 1932, **11**, 140) and a procedure for the micro-determination of active hydrogen in organic compounds with $LiAlH_4$ in di-*n*-butyl ether are described. The applicability of the method is limited to compounds soluble in di-*n*-butyl ether since no separate solvent is used for dissolving the substance. I. JONES

3007. **A new method for the determination of oxygen-18 in organic compounds.** H. Dahn and H. Moll (Univ. Basel, Switzerland). *Chem. & Ind.*, 1959, (12), 399–400.—*o*-Phenylenediamine hydrochloride (**I**) reacts with aldehydes, ketones, acids and amines to give either a Schiff's base or benzimidine and H_2O; the H_2O can be equilibrated with inactive CO_2 for mass-spectrometric isotopic analysis. The method was tested without labelling the organic compounds. The unlabelled compound was heated with **I** and labelled CO_2 of known tracer abundance (1·1% of ^{18}O in excess), the dilution of the ^{18}O in the CO_2 giving a measure of the quantity of H_2O formed. Recoveries from 25 compounds were > 95%. G. P. COOK

3008. **Determination of metals in organic compounds by the closed flask method.** R. Belcher, A. M. G. Macdonald and T. S. West (Dept. of Chem., Birmingham Univ., England). *Talanta*, 1958, **1** (4), 408–410.—The method of combustion in O in a closed flask, previously used for halogens and S (*cf.* Schöniger, *Anal. Abstr.*, 1955, **2**, 1816; 1957, **4**, 924), has been applied to the determination of metals. Of those studied, Zn, Cd and Mg can be determined by absorbing the combustion products from 5 to 10 mg of sample in *N* HCl (5 ml), neutralising with aq. NH_3, adding a specified aq. NH_3 - NH_4Cl buffer (10 ml) and excess of 0·005 *M* EDTA (10 ml) and back-titrating with 0·005 *M* $MgCl_2$, with Solochrome black 6B as

indicator. Oxides of nitrogen are removed by boiling with sulphamic acid, and iodine with $NaHSO_3$ and formaldehyde. Boron is determined by absorption in water, and titrating with alkali in the presence of mannitol. The method is less satisfactory for As than those already available, some As being lost by alloying with the platinum-wire sample-holder; Pb, Bi and probably Sb also form alloys. Some metals (*e.g.*, Ni and Ga) form oxides which cannot be dissolved by solvents suitable for EDTA determinations. Alkali and alkaline-earth metals might be determinable by flame spectrophotometry. Although promising, the method is not yet generally applicable.

R. E. ESSERY

3009. **Microchemical reactions of sulphur compounds.** H. M. Romijn. *Pharm. Weekbl.*, 1958, **93** (21), 929–931.—Many organic sulphur compounds can be identified by the reaction between 1 to 2 mg of the sample and an equal quantity of a metal acetate in 1 drop of 10% aq. NH_3. The appearance of crystalline ppt. formed with Cu acetate and Co acetate, and, in certain cases, with other acetates, is described in tabular form.

G. BURGER

3010. **Identification of higher *n*-alkyl groups in alicyclic and aromatic hydrocarbons by oxidation with chromic acid.** M. Jureček, M. Souček, J. Churáček and F. Renger (Chem.-Technol. Hochschule, Pardubice, ČSR). *Z. anal. Chem.*, 1959, **165** (2), 109–118.—The method is based on the oxidation of the higher *n*-alkyl groups, present as side-chains in alicyclic and aromatic hydrocarbons, with chromic and sulphuric acids giving a mixture of fatty acids. These are steam-distilled and converted into the ethylammonium salts, which are separated by paper chromatography. The mobile phase is *n*-butanol saturated with 0·1 *N* aq. ethylamine; the individual ethylammonium salts are identified with ninhydrin soln. The procedure was applied to 1-alkyl*cyclo*hexenes, alkyl*cyclo*hexanes (alkyl groups C_1 to C_4) and alkylbenzenes (alkyl groups C_2 to C_7).

G. P. COOK

3011. **Determining molecular structure by nuclear magnetic resonance of hydrogen.** N. F. Chamberlain (Res. and Devel. Dept., Humble Oil and Refining Co., Baytown, Tex.). *Anal. Chem.*, 1959, **31** (1), 56–77.—A rapid and accurate method for determining structural features of hydrocarbons and their derivatives in pure form or in mixtures is presented. By this technique a variety of organic functional groups, under a wide variety of conditions, can be identified quickly and positively without precise calibration with the exact compounds being studied. The spectra of over 400 pure compounds have been obtained under standard reproducible conditions, and the chemical shift information is summarised in a series of charts.

K. A. PROCTOR

3012. **Quantitative determination of gases by chromatography.** H. Pietsch (Dtsch. Erdöl A.-G., Heide, Holstein). *Erdöl u. Kohle*, 1958, **11** (10), 702–705.—The separation of the components of a mixture of H and hydrocarbons with boiling-points up to that of *cis*but-2-ene (3·72°) is described. Hydrogen and methane are separated by adsorption chromatography on silica gel, with CO_2 as carrier gas. The relative concn. are determined from a graph of time *vs.* vol. The remaining components are separated by partition chromatography in an apparatus in which a thermal-conductivity cell is used as indicator. If the peaks are sharply separated, the relative concn. can be determined from the peak height. The peak heights are sensitive to many factors, especially temp., and an internal standard is necessary. By assuming the peaks to be symmetrical, the relative concn. can be determined, even when the peaks are not sharply separated, by measurement of the area beneath the peaks. The peak areas are proportional to wt. % rather than mol. %. As carrier gases, H and He give the greatest sensitivity. A correction, which is valid only for very low concn. of the sample in the carrier gas, must be made for the variation of thermal conductivity with temp. The procedure is suitable for the routine analysis of refinery gases.

G. BURGER

3013. **Analysis of hydrocarbons.** H. M. Frey (Univ. Southampton, England). *Nature*, 1959, **183**, 743–744.—A mixture of ethane, ethylene, propane and propylene can be separated by gas chromatography on a single column, packed with acetylacetone on fire brick and operated at 0°. The column also resolves almost all the isomers of the hydrocarbons up to C_4, including many cyclic compounds. Separation of *iso*butylene and but-1-ene in the presence of other hydrocarbons is also achieved. 1:1-Dimethyl*cyclo*propane and 3-methylbut-1-ene are easily separated by a soln. of $AgNO_3$ in ethanediol on fireclay; this column can be used in conjunction with others.

G. P. COOK

3014. **Gas-chromatographic analyses of very pure ethylene.** G. Nodop (Esso A.-G., Raffinerie Köln, Germany). *Z. anal. Chem.*, 1958, **164** (1), 120–127.—The determination of trace impurities (H, O, N, CO, CH_4, CO_2, C_2H_6, and C_3 and C_4 hydrocarbons) by using tetra*iso*butylene, molecular sieve 13X and silica gel as stationary phases is described. The results are tabulated.

B. B. BAUMINGER

3015. **Methods of analysis of gas mixtures containing acetylene.** W. Hunsmann (Untersuchungslab. der Chem. Werke Hüls A.-G., Germany). *Z. anal. Chem.*, 1958, **164** (1), 57–61.—Acetylene compounds have relatively simple and characteristic mass spectra but there is uncertainty in the hydrogen determination. The normal pressure method, an extended form of the Orsat analysis, has given reasonable results for paraffins containing 2, 3 or 4 C atoms, olefins and acetylene. The pptn. method with ammoniacal $AgNO_3$ soln., combined with mass spectrometry, has been examined and the data are tabulated. It is shown that no method by itself leads to a precise and complete result in the analysis of complicated gas mixtures.

B. B. BAUMINGER

3016. **Selective reactivity in gas - liquid chromatography. Determination of 2-bromobutane and 1-bromo-2-methylpropane.** W. E. Harris and W. H. McFadden (Chem. and Metallurgy Div., Atomic Energy of Canada Ltd., Chalk River, Ontario, Canada). *Anal. Chem.*, 1959, **31** (1), 114–117.—Separation of 2-bromobutane (**I**) and 1-bromo-2-methylpropane (**II**) from other alkyl bromides is performed by conventional chromatography on either Tween 60 or dimethylsulpholane with He as carrier gas. To determine **II** alone, allow the sample to pass through a reaction tube packed with successive zones of $AgNO_3$ on fire-brick (**III**), **III**, H_2SO_4 on **III**, **III**, and Na_2HPO_4

on **III** before it enters the chromatographic column. The $AgNO_3$ removes **I** and other secondary and tertiary alkyl bromides, but not **II** and other primary bromides. The H_2SO_4 and Na_2HPO_4 remove the butenes and HNO_3 which are produced by the decomposition of **I**. As little as 0·5% of **II** can be detected in a mixture (6 μl) containing 99·5% of **I**. **I** is determined indirectly, and the sensitivity is less. A. R. ROGERS

3017. **Paper chromatography of organic peroxides.** A. Rieche and M. Schulz (Inst. Org. Chem., Dtsch. Akad. Wissenschaft., Berlin-Adlershof). *Angew. Chem.*, 1958, **70** (22–23), 694–696.—Organic hydroperoxides and peroxides are separated by inverse-phase chromatography on acetylated paper (S. & S. 2043b) with ethyl acetate - dioxan - water (2·0: 4·5: 4·6) as solvent. The spots are made visible by spraying with *p*-aminodimethylaniline hydrochloride soln. (3%) or KI soln. (3%) in aq. methanol. Dialkyl and diacyl peroxides do not react with either of these reagents unless they are hydrolysed on the developed chromatogram by spraying with conc. HCl. R_F values are given for H_2O_2 and a number of organic peroxides. W. T. CARTER

3018. **Iodimetric determination of organic hydroperoxides.** H. Hock and H. Kropf (Inst. f. Brennstoffchem., Bergakademie Clausthal, Harz, Germany). *Chem. Ber.*, 1959, **92** (5), 1115–1117.—The hydroperoxide (10 to 50 mg) is dissolved in acetic acid (10 ml) and after the addition of a little solid CO_2 (for removal of atmospheric O) is treated with 0·5 ml of 50% KI soln. and a granule of cuprous chloride. After 5 min. the soln. is diluted with water (25 ml) and titrated with 0·01 *N* thiosulphate. Satisfactory results are given for cumyl hydroperoxide. N. E.

3019. **Identification of alcohols by microscopic mixed fusion analysis.** D. E. Laskowski and O. W. Adams (Dept. of Chem. and Chem. Engng, Armour Res. Foundation, Illinois Inst. Technol., Chicago). *Anal. Chem.*, 1959, **31** (1), 148–152.—The 2:4:6-trinitrobenzoates of alcohols are fused with either naphthalene or phenanthrene and identification is made from the significant m.p. of the fused molecular addition compound (*cf.* Laskowski *et al.*, *Anal. Abstr.*, 1954, **1**, 6; 1958, **5**, 3040). A. R. ROGERS

3020. **Separation of carbohydrate derivatives by gas - liquid partition chromatography.** A. G. McInnes, D. H. Ball, F. P. Cooper and C. T. Bishop (Nat. Res. Council, Ottawa, Canada). *J. Chromatography*, 1958, **1** (6), 556–557.—The O-methyl derivatives of the methyl glycosides of the heat-labile monosaccharides are sufficiently stable and volatile to be analysed by gas - liquid partition chromatography. Several fully methylated methyl glycopyranosides were sufficiently separated to allow quantitative determination of their mixtures. G. P. COOK

3021. **New indirect method for the titration of glucose using sodium cuprithiosalicylate.** C. Rabega. *An. Univ. "C. I. Parhon", Bucureşti, Ser. Ştiinţ. Nat.*, 1956, (12), 57–63.—An aq. soln. of Na cuprithiosalicylate (**I**) became turbid when heated and after the addition of NaOH turned black owing to the formation of CuO. If reducing substances were present, Cu_2O was formed, and the degree of formation of Cu_2O could be used to determine the amount of reducing substances present. By using the iodimetric method of Haen and Low, the amount of Cu^{2+} was determined in 10 ml of a 2% soln. of **I**, alone or in the presence of increasing amounts of glucose (2 to 10 mg). The amount of Cu^{2+} decreased linearly with the increase in the amount of glucose; Cu^{+} also increased linearly. A calibration curve was constructed and the amount of glucose in an unknown sample determined from it. This method is more rapid than the Bertrand method and can be used to determine much smaller amounts. SUGAR IND. ABSTR.

3022. **Indirect micro-determination of fructose and inverted sucrose with sodium cuprithiosalicylate.** C. Rabega, M. Rabega and R. Stănescu. *An. Univ. "C. I. Parhon", Bucureşti, Ser. Ştiinţ. Nat.*, 1957, (16), 111–116.—A standardised 2% soln. of Na cuprithiosalicylate has been used to determine the amount of fructose in soln. containing 2 to 10 mg of fructose and also in soln. containing mixtures of fructose and glucose resulting from the inversion of sucrose. The procedure involves the Haen and Low method for the iodimetric titration of Cu^{2+} left in 10 ml of the reagent in the presence of various amounts of sugar, and can be used for the indirect micro-determination of inverted sucrose. The amount of sucrose is calculated from the invert sugar content by multiplying by a factor of 0·95. The method is very exact because the experimental errors are almost negligible. The time for one micro-determination is about 7 min. SUGAR IND. ABSTR.

3023. **New method for the micro-titration of certain reducing mono- and di-saccharides by using cuprithiosalicyate.** C. Rabega, R. Stănescu, M. Rabega and T. Osorhan. *An. Univ. "C. I. Parhon", Bucureşti, Ser. Ştiinţ. Nat.*, 1956, (12), 65–75.—The Rabega method (*cf. Anal. Abstr.*, 1959, **6**, 3021) of titrating CuO for determining glucose is used to determine mannose, galactose, maltose and lactose. SUGAR IND. ABSTR.

3024. **Rapid detection of sorbitol in presence of other polyhydric alcohols by means of one-dimensional paper chromatography.** G. Cerutti and I. Trabucchi (Ist. Chemioterapico Italiano, Milan). *Chim. e Ind.*, 1958, **40** (10), 819–821.—Sorbitol is separated from other polyhydric alcohols (except mannitol) by paper chromatography by using as mobile phase *n*-butanol saturated with H_2O, or *n*-butanol - acetic acid - H_2O (4:1:5) (non-aq. layer), the spots being revealed with $FeCl_3$ and ascorbic acid. Pure sorbitol gives only a single spot, but non-cryst. sorbitols also give spots characteristic of anhydro derivatives and other unidentified substances. L. A. O'NEILL

3025. **Quantitative oxidation of ammonium compounds and formaldehyde by bromine (bromate - bromide) in acid solution.** D. Kőszegi and E. Salgó (Pharm. Chem. Inst., Med. Univ., Szeged, Hungary). *Z. anal. Chem.*, 1958, **164** (3), 320–327.—The bromatometric procedure is extended to the determination of hexamine, formaldehyde and inorg. ammonium compounds [NH_4Cl, NH_4NO_3 and $(NH_4)_2SO_4$, with various amounts of NaCl]. It is based on the oxidation of these compounds in HCl soln. with 0·1 *N* $KBrO_3$ and the iodimetric titration of the excess of $KBrO_3$. One determination takes approx. 35 min. Details of the procedure are given. B. B. BAUMINGER

3026. **Simple sensitive test for aliphatic ketones.** E. Sawicki and T. W. Stanley (Robert A. Taft Sanitary Engng Center, U.S. Public Health Service, Dept. of Health, Education and Welfare, Cincinnati 26, Ohio). *Anal. Chem.*, 1959, **31** (1), 122–124.—Add a soln. of the sample in 2-methoxyethanol (0·5 ml) to a mixture of 2-hydroxy-1-naphthaldehyde - $AlCl_3$ (1:3) (75 mg) and set aside for 3 min. A stable blue colour, or with some ketones a red or violet colour, is given by 1 to 5-μg quantities of almost all aliphatic ketones which contain the structure $-CH_2{\cdot}CO{\cdot}CH_2-$. A blank test is advisable for comparison. A. R. ROGERS

3027. **Study of the infra-red spectra of some β-diketones.** M. C. de Wilde-Delvaux and P. Teyssié (Univ. of Louvain, Belgium). *Spectrochim. Acta*, 1958, **12** (4), 289–293 (in French).—The i.r. spectra of methacroylacetone and its polymer, 2-heptyl-, 2-heptyl-4-methyl-, 2-pentyl- and 4-methyl-*cyclo*hexane-1:3-dione in the C=O and C=C stretching region are discussed and compared with that of acetylacetone. A study of the keto - enol equilibrium shows it to be a function of dilution for the derivatives of *cyclo*hexane-1:3-dione, indicating intermolecular hydrogen bonding, but for the other examples it is a function of the polarity of the solvent. The doublet form of the C=O stretching band is attributed to interaction between the two C=O groups on the same C atom. E. G. CUMMINS

3028. **Infra-red absorption study of *n*-alkylmalonic acids and their diethyl esters.** K. Nagaraja Rao and A. B. Biswas (Nat. Chem. Lab., Poona, India). *Spectrochim. Acta*, 1958, **13** (1–2), 151–153.—The i.r. absorption spectra of amylmalonic acid and its diethyl ester are illustrated, and their differences discussed. Results are also tabulated for a homologous series of *n*-alkylmalonic acids and the corresponding saturated fatty acids, and discussed with respect to structural considerations. B. B. BAUMINGER

3029. **Determination of alginic acid.** Shingo Miyake (Inst. Sci., Tokyo Univ., Yayoi-cho, Chiba). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1958, **61** (10), 1278–1281.—Since alginic acid (**I**) is adsorbed neither by anion- nor by cation-exchange resin, ionic impurities in commercial sodium alginate (< 0·01 *N*) are completely removed by successive treatment with these resins, **I** in the percolate being titrated conductimetrically or potentiometrically with 0·1 *N* NaOH. For dry **I**, which is not readily soluble in water, the method is unsuitable. The results agree well with those by McCrady's CO_2 evolution method (*cf. Ind. Eng. Chem., Anal. Ed.*, 1946, **18**, 190). *Procedure*—Dissolve the sample (0·5 g) in water (250 ml), filter and pass the filtrate through a column (15 mm × 160 mm) of Amberlite IRA-410. Discard the first 60-ml fraction and pass the succeeding fraction through a column (9 mm × 150 mm) of Amberlite IR-120. Discard the first 40-ml fraction and titrate the following 50-ml fraction with NaOH soln. K. SAITO

3030. **Mass-spectrometric studies. I. Methyl esters of saturated normal-chain carboxylic acids.** R. Ryhage and E. Stenhagen (Kem. Inst. I, Karolinska Inst., Stockholm). *Ark. Kemi*, 1959, **13** (6), 523–542.—Data are given of the mass spectra of a homologous series of methyl esters of saturated normal-chain carboxylic acids up to and including methyl *n*-hexacosanoate.

3031. **Mass spectra of esters. Formation of rearrangement ions.** A. G. Sharkey, jun., J. L. Shultz and R. A. Friedel (Bureau of Mines, Region V, U.S. Dept. of the Interior, Pittsburgh, Pa., U.S.A.). *Anal. Chem.*, 1959, **31** (1), 87–93.—Rearrangement and normal fragmentation ions have been studied in the mass spectra of 31 esters, ranging from methyl formate to decyl acetate. Both types of ions are useful in identifying esters. Rearrangement peaks can be used for the qual. determination of the distribution of the products of Fischer - Tropsch syntheses. K. A. PROCTOR

3032. **The ultra-violet absorption spectrum of dicyanoacetylene.** F. A. Miller and R. B. Hannan, jun. (Univ. of Pittsburgh, Pa., U.S.A.). *Spectrochim. Acta*, 1958, **12** (4), 321–331.—The spectrum of the soln. consists of four prominent bands below 270 mμ and one weak one at 286 mμ. The spectrum of the vapour, measured at 30 and 90 mm of Hg in a 5-mm cell, shows a hypsochromic shift of 870 cm^{-1} and each of the four prominent bands becomes six bands. Further experiments aimed at higher resolution reveal the detail of the two band systems. The more intense one begins near 268 mμ and extends towards lower wavelengths. It consists of the four well-spaced groups of bands with six major bands in each group. The other group, centred near 280 mμ, features six bands. The measured frequencies of 35 bands are tabulated. Fundamental vibration frequencies are listed and the postulation of a $^1\Sigma_g^+ \rightarrow {}^1\Sigma_u^-$ transition for the 280-mμ and a $^1\Sigma_g^+ \rightarrow {}^1\Delta_u$ transition for the 268-mμ band system is discussed. It is concluded that this latter "electronically forbidden" transition may be "vibrationally allowed". E. G. CUMMINS

3033. **Colour reaction of 2-(3-oxo-1-indanylidene)-indane-1:3-dione. Micro-determination of aliphatic amines in potassium salt concentrates.** A. Singewald (Wintershall A.-G., Werk Neuhof bei Fulda, Germany). *Z. anal. Chem.*, 1958, **164** (2), 219–232.—A new method is described for the quant. determination of microgram amounts of primary aliphatic amines, particularly those employed in technical potassium salt flotation. It does not depend on a specific reaction between the amine and 2-(3-oxo-1-indanylidene)indane-1:3-dione, but on a tautomeric effect regulated by pH. A two-phase system is used; amines that display a so-called apparent basicity in the organic phase induce a change to the enol form of the reagent and give a colour in the organic layer, which depends on the amount of amine present and obeys Beer's law. Inorganic salts are held in the aq. layer, where a buffer soln. prevents their interference. Modified forms of the reagents are discussed. *Procedure*—To 5 g of KCl containing an amine hydrochloride add 5 ml of phosphate - citrate buffer (pH 6·5 to 7·0) and 15 ml of ethereal reagent soln. (0·2 g per litre). Make up to 50 ml with H_2O, separate the ethereal layer and measure the extinction at 510 to 545 mμ. P. D. PARR-RICHARD

3034. **Determination of biuret in urea by ion-exchange resins.** T. Takahashi and D. Yoshida (Hatano Tobacco Exp. Sta., Japan). *Soil and Plant Food, Tokyo*, 1958, **3**, 142–144.—The sample of urea, containing about 2 mg of biuret, is dissolved in 100 ml of warm water. A 25-ml aliquot is passed through a column of Amberlite resin, IRA-400AG, having a vol. of about 5 ml and length 60 mm. The column is washed free from

urea with water and the biuret is eluted by 100 ml of 2 *N* HCl and determined by the micro-Kjeldahl method. With compound fertilisers, other anions may interfere with the adsorption of biuret on the column. To avoid this, the fertiliser (10 g) is extracted with anhyd. methanol (100 ml) in a Soxhlet apparatus for 2 hr. In some instances extraneous anions may occur in the methanol extract and are removed by passage of the extract through Amberlite IR-4B. Methanol has no detrimental effect on the determination. In the early stages of use the resin IRA-400AG may release some N during elution and "blank" tests are advised. A. G. POLLARD

3035. **Infra-red spectra of some methylammonium iodides: angle deformation frequencies of $\overset{+}{N}H$ and $\overset{+}{N}H_2$ groups.** E. A. V. Ebsworth and N. Sheppard (Univ. Chem. Lab., Lensfield Road, Cambridge, England). *Spectrochim. Acta*, 1959, **13** (4), 261–270.—A study has been made of the infra-red spectra of di-, tri- and tetra-methylammonium iodides, and of N-deutero samples of di- and tri-methylammonium iodides. The samples were in the form of mulls in Nujol and hexachlorobutadiene, and spectra were recorded in the range 4000 to 400 cm^{-1}. The observed absorption frequencies are assigned to various modes of vibration (the deuterated samples being of assistance here), and frequencies are compared with those of the isoelectronic hydrocarbons $(CH_3)_2CH_2$, $(CH_3)_3CH$ and $(CH_3)_4C$. Superior spectra are claimed for the iodides, these being less hygroscopic than the chlorides. H. M.

3036. **Infra-red spectra of organic thiocyanates and *iso*thiocyanates.** E. Lieber, C. N. R. Rao and J. Ramachandran (Dept. of Chem., De Paul Univ., Chicago, Ill., U.S.A.). *Spectrochim. Acta*, 1959, **13** (4), 296–299.—A study has been made of several organic thiocyanates and *iso*thiocyanates and the important absorption frequencies are tabulated. The 2140 cm^{-1} band of the thiocyanates and the 2060 cm^{-1} to 2105 cm^{-1} band of the *iso*thiocyanates are suggested as suitable for identifying such compounds singly or in mixtures. H. M.

3037. **Determination of alkyl phosphites in the presence of other organic phosphorus compounds.** A. Yarden and C. Eger. *Bull. Res. Council Israel, A*, 1958, **7**, 80–81.—To a weighed amount of the mixture (containing 50 to 150 mg of dialkyl phosphite) add 10 ml of a 0·2 *N* soln. of iodine in pyridine, keep tightly stoppered for 30 min. in the dark, then add 20 ml of peroxide-free dioxan, followed by 5 *N* HCl (24 ml), in small amounts, with cooling. Titrate the soln. with 0·1 *N* $Na_2S_2O_3$ until the colour changes to yellow. Add 75 ml of water and 1% starch soln. (5 ml) and complete the titration. The difference between this result and a blank experiment is taken. The results are reproducible, but not stoicheiometric, therefore a reference curve has to be prepared with known amounts of pure phosphite. CHEM. ABSTR.

3038. **Water content of tributyl phosphate by high-resolution nuclear magnetic resonance.** B. B. Murray and R. C. Axtmann (Savannah River Lab., E. I. du Pont de Nemours & Co., Inc., Aiken, S.C.). *Anal. Chem.*, 1959, **31** (3), 450–451.—The method involves the measurement of the chemical shift of the water proton resonance with respect to the methyl proton resonance in tributyl phosphate and thus requires no external standard. The method is non-destructive and requires only 2 min. per determination once the spectrometer is set up. The precision of individual measurements is equiv. to 0·13% (w/w) of water and is not dependent upon gain stability and linearity in the instrument.

K. A. PROCTOR

3039. **Analysis of products containing *cyclo*pentadiene, with special reference to gas chromatography.** E. A. M. F. Dahmen and J. D. van der Laarse (Koninklijke Shell-Lab., Amsterdam, Holland). *Z. anal. Chem.*, 1958, **164** (1), 37–48.—A method of de-polymerisation of crude *cyclo*pentadiene products is described and the results of the gas-chromatographic analysis are discussed.

B. B. BAUMINGER

3040. **The ultra-violet and infra-red spectra of the methylbiphenyls: a preliminary analysis.** G. H. Beaven and E. A. Johnson (Nat. Inst. Med. Res., Mill Hill, London). *Spectrochim. Acta*, 1959, **14**, 67–81.—The effects of progressive substitution in the four sterically sensitive *ortho* groups are studied in the work described. The coplanarity condition for max. conjugation between aromatic groups, and the electron diffraction evidence that the 1:1′ biphenyl bond has only 10% double-bond character inspire the theoretical argument. Tabulated results show that a single or two symmetrical methyl substituents at the 3- or 4-positions have little special effect. In the 2- (*ortho*) position, however, a single methyl group causes a marked reduction in intensity and a blue shift of the conjugated band accompanied by the appearance of a poorly resolved low-intensity long-wave inflection. The 2:2′-dimethylbiphenyl shows the conjugated band further shifted to shorter wavelength as an unresolved inflection heavily overlapped by an intense band system at about 200 mμ, while the long-wave absorption feature is now a definite band of low intensity, with characteristic, just resolved, benzenoid fine structure. Results from asymmetrical and further symmetrical substitutions are presented and discussed. Some support is found for the theory that electronic transitions involving both phenyl groups in biphenyls can occur by an interaction process that is apparently independent of the coplanarity condition. The infra-red work included shows, in some cases, a band at 1060 cm^{-1} which is significant for biphenyl itself, as distinct from other bands which must be attributable to substituted benzene ring absorption.

E. G. CUMMINS

3041. **Quantitative analysis of multi-components. I. Gas-chromatographic examinations of phenol mixtures.** G. Bergmann and D. Jentzsch (Inst. f. Spektrochem. u. angew. Spektroskopie, Dortmund, Germany). *Z. anal. Chem.*, 1958, **164** (1), 10–29.—On etherification of the phenol group, before gas-chromatographic separation, the polar character of the phenol is reduced, the temp. of boiling is lowered, and the interaction between the substance and the stationary phase in the column changed. The behaviour of the single components at different working temp., and with various stationary phases with regard to intermolecular forces, is discussed. The reproducibility of gas-chromatographic analyses of phenol mixtures is tabulated.

B. B. BAUMINGER

3042. **Gas - liquid chromatographic separation of phenols as trimethylsilyl ethers.** S. H. Langer, P. Pantages and I. Wender (U.S. Bur. of Mines, Pittsburgh, Pa., U.S.A.). *Chem. & Ind.*, 1958, (50), 1664–1665.—The almost complete conversion of coal-tar phenols into their trimethylsilyl ethers by heating under reflux for $\simeq$ 16 hr. with hexamethyldisilazane (*cf.* Langer *et al.*, *Anal. Chem.*, 1958, **30**, 1353) permits the use of a wider variety of liquid phases for the gas - liquid chromatographic separation of phenols. Relative retention vol. at 125° for di-*n*-butyl tetrachlorophthalate and D.C. Silicone 550 are listed for the trimethylsilyl ethers of 15 phenols, and the effective separation, on a column (366 cm × 0·5 cm), of seven phenols in a sample ($\simeq$ 3·5 μl) of commercial cresol by this method is outlined. The ethers of *o*-, *m*- and *p*-cresols are eluted in the order of their b.p. (194° to 199°). Concn. of aq. phenol as low as 0·2%, and the presence of such compounds as guaiacol or catechol in the original sample, can be readily detected. W. J. BAKER

3043. **Quantitative paper-chromatographic determination of phenols.** R. W. Keith, D. Le Tourneau and D. Mahlum (Univ. Idaho, Moscow, U.S.A.). *J. Chromatography*, 1958, **1** (6), 534–536.—The samples are dissolved in 50% ethanol and spots (10 to 100 μg) are applied to Whatman paper (No. 1, 4 or 54); spots of known phenolic compounds are applied on either side of the main band containing the unknown. The papers are then irrigated by using the descending technique at room temp. After drying, the standard spots are located with Folin - Ciocalteu reagent (**I**) and the corresponding areas of the unknown spots are cut from the paper and the phenols are eluted and determined with **I**, the intensities being measured at 660 mμ. R_F values for the common phenols with the solvent systems benzene - acetic acid - water (2:2:1), 20% acetic acid, and ethyl acetate - acetic acid - water (2:1:2) are listed. G. P. COOK

3044. **Separation of mono- and di-hydric phenols by gas chromatography.** J. Janák and R. Komers (Lab. for Gas Analysis, Czech Acad. of Sci., Brno). *Z. anal. Chem.*, 1958, **164** (1), 69–72.—Selective packing of columns (*e.g.*, with Apiezon L, phenylmercaptothiadiazole, erythritol, dulcitol) is used to separate mono- and di-hydric phenols, and the results are discussed and tabulated. B. B. BAUMINGER

3045. **Inverse isotope dilution analysis of salicylic acid.** H. A. Swartz and J. E. Christian (Bionucleonics Lab., School of Pharmacy, Purdue Univ., Lafayette, Ind.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1958, **47** (10), 701–702.—Prepare a soln. of the sample (containing 20 to 200 mg of salicylic acid) in glacial acetic acid (**I**). Add a known quantity in excess of ^{131}ICl of known specific activity dissolved in **I**. Heat slowly to 70° or 80° and maintain at that temp. for 20 min., with agitation. Add a known quantity in large excess of unlabelled 3:5-di-iodosalicylic acid, stir and set aside the soln. to cool. Collect the crystals, wash with 20% aq. **I** and with H_2O, dry and re-crystallise in turn from acetone, **I** and acetone again. Dry and weigh the crystals, dissolve in **I** and determine the radioactivity. The procedure may be applied to the analysis of acetylsalicylic acid after quant. hydrolysis to salicylic acid. A. R. ROGERS

3046. **Polyhydroxy (catecholic) phenolic acids—the formation of *m*-hydroxy and methoxy derivatives in man.** S. L. Tompsett (Biochem. Lab., Northern General Hospital, Edinburgh). *J. Pharm. Pharmacol.*, 1959, **11** (1), 32–35.—A method is given for the determination of methoxyl groups in phenolic compounds; N-methyl groups do not interfere. *Procedure*—Heat the sample (containing $\simeq$ 1 mg of methoxyl compound) to fuming with H_2O (10 ml) and conc. H_2SO_4 (5 ml) and collect the distillate. Allow to cool, add H_2O (5 ml) and redistil; repeat this part of the procedure. Mix $NaHCO_3$ (1 g) with the combined distillates and heat, and to the first 10 ml of distillate add 5% aq. ethanol (1 ml), 1% $KMnO_4$ soln. (5 ml) and 25% H_3PO_4 soln. (1 ml). Allow to stand for 1 hr. at room temp. with frequent shaking, remove the excess of permanganate by addition of H_2O_2 soln., heat to boiling and collect the first 10 ml of distillate. Dilute an aliquot to 3 ml with H_2O, add chromotropic acid reagent (5 ml), heat in a bath of boiling water for 30 min., cool, dilute to 10 ml with 9 *N* H_2SO_4 and measure the extinction at 570 mμ against a reagent blank. A. R. ROGERS

3047. **Gas chromatography of aromatic amines and nitro compounds.** J. H. Jones, C. D. Ritchie and K. S. Heine, jun. (Div. Cosmetics, Food and Drug Admin., Dept. of Health, Educ. and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (4), 749–752.—The column employed is a copper tube containing Firebrick C–22 impregnated with DC710 silicone fluid; the detector is a thermal-conductivity cell and recorder. The sample inlet is heated at 260°; analyses are made at a flow rate of He of 20 ml per min., and a column temp. of 200° to 210°. Retention times of various isomeric amino- and nitro-substituted hydrocarbons are tabulated. Satisfactory analyses of mixtures of *m*-xylidine, *o*-xylidine, mesidine, ψ-cumidine and *iso*duridine are recorded. A. A. ELDRIDGE

3048. **Spot-test detection of phenylhydrazine and arsonic acids.** F. Feigl and E. Jungreis (Lab. da Prod. Mineral, Min. da Agric., Rio de Janeiro, Brazil). *Talanta*, 1958, **1** (4), 367–368.—When phenylhydrazine or its salts are heated with arsenic acid or aliphatic or aromatic arsonic acids, phenol is liberated which is detected by the indophenol reaction with 2:6-dichloro-*p*-benzoquinone-4-chlorimine (**I**). *Detection of phenylhydrazine*—Place a drop of test soln. in a micro test-tube with a drop of a conc. soln. of arsenic acid. Cover the mouth of the tube with a paper impregnated with a saturated benzene soln. of **I**. Heat in a boiling-water bath for 3 to 4 min. A positive reaction is shown by a pale-brown stain on the paper, which turns blue when exposed to NH_3. The blue is not permanent, but can be restored by re-exposure to NH_3. The limit of detection is 5 μg. Phenylhydrazones and phenylosazones can also be detected. If phenols are present they must be removed by extraction of the alkaline test soln. with an immiscible solvent, the phenols remaining in the aq. layer. *Detection of arsonic acids*—Place a little of the solid, or a drop of its aq. soln., in a micro test-tube with a further drop of water and several centigrams of phenylhydrazine hydrochloride. The reagent paper is placed over the mouth of the tube, which is heated in boiling water for 5 min., and the paper is exposed to NH_3 as before. A blue stain indicates arsonic acid. The limits of detection

are 5 μg for *p*-amino- and *p*-hydroxy-phenylarsonic acids, and 10 μg for *p*-dimethylaminophenylazophenylarsonic acid. R. E. ESSERY

3049. **Quantitative determination of hydrazobenzene with alkaline permanganate.** R. Reiss (Anal. Lab., Fa. Byk-Gulden Lomberg, Konstanz am Bodensee). *Z. anal. Chem.*, 1958, **164** (4), 402–407.—From 10 to 140 mg of hydrazobenzene in xylene soln. can be quant. oxidised to *trans*-azobenzene by being shaken for ≃ 10 min. with 0·1 *N* NaOH (30 ml) and 0·1 *N* $KMnO_4$ (25 ml). After addition of KI (1 g) and 25% H_2SO_4 (20 ml), the liberated iodine is titrated with 0·1 *N* $Na_2S_2O_3$ and the titre (ml) is subtracted from 25 ml (the vol. of $KMnO_4$ soln. added). The max. error is ≃ ±1 mg, and the method is recommended for the analysis of commercial hydrazobenzene. Aniline and phenylhydroxylamine interfere, but azo-, azoxy-, nitro- and nitroso-benzene do not. W. J. BAKER

3050. **Chromatographic analysis of commercial mixtures of nitro compounds.** J. Pospíšil and V. Ettel (Inst. Org. Technol., High School of Chem. Technol., Prague). *Chem. Průmysl*, 1958, **8** (10), 511–515.—The equations for the calculation of R_F values formulated on the basis of the experimental results of Franc *et al.* (*Chem. Listy*, 1955, **49**, 317, 325, 328, 872; 1956, **50**, 373, 547) are important for studying the chromatographic behaviour of organic compounds and can also be used for the identification of components of various mixtures. The mixture must first be separated into fractions containing compounds with similar functional groups. By this method the probable presence, in a single spot, of components with similar R_F values can be detected and a suitable solvent system selected. The relation between R_F and dipole moment for the fixed polar or non-polar phases is also valid under certain conditions for unimpregnated and acetylated papers. The method has been tested by separating mixtures of nitro compounds. The separation has been carried out on Whatman paper No. 1 and 4 with fixed polar phase, on unimpregnated paper, on paper with a fixed non-polar phase and on acetylated paper. R_F values are tabulated for 12 mono-, di- and trinitro-benzenes, -toluenes and -phenols. J. ZÝKA

3051. **Constant-potential coulometric reduction of organic nitro and halogen compounds.** V. B. Ehlers and J. W. Sease (Dept. of Chem., Wesleyan Univ., Middletown, Conn.). *Anal. Chem.*, 1959, **31** (1), 16–22.—Constant-potential coulometric reduction at a mercury-pool cathode has been tested with 18 organic nitro compounds and 8 organic halogen compounds. Methanolic LiCl or methanolic tetraethylammonium bromide was used as solvent. After correction for the residual current and for incomplete reduction of the sample compound, the error was usually < 1% at sample concn. of ≮ 5×10^{-4} *M*. Two-component mixtures could be analysed if the reduction waves were separated by ≮ 0·35 V. A. R. ROGERS

3052. **Use of paper chromatography for analysis and identification of naphthylamine- and naphthol-sulphonic acids.** T. M. Selezneva (M. V. Frunze Dorogomilovsk Chem. Works). *Zavod. Lab.*, 1958, **24** (11), 1353–1356.—Paper-chromatographic methods for the control of various stages in the production of 6-amino- and 7-amino-1-naphthol-3-sulphonic acids are described. G. S. SMITH

3053. **Polarographic determination of 1-diazo-2-naphthol-4-sulphonic acid and of 1-diazo-6-nitro-2-naphthol-4-sulphonic acid.** Kh. Budevska. *Compt. Rend. Acad. Bulgare Sci.*, 1958, **11** (5), 387–390.—For 1-diazo-2-naphthol-4-sulphonic acid (**I**), dissolve the dry substance (0·3 g) or the wet paste (0·6 g) in H_2O (100 ml). To a 10-ml aliquot add aq. NH_3 - NH_4Cl buffer soln. (25 ml) at pH 9 and 0·5% gelatin soln. (5 ml). Make up to 100 ml. Polarograph the soln. For 1-diazo-6-nitro-2-naphthol-4-sulphonic acid (**II**), dissolve the wet paste (0·6 g) in H_2O (100 ml). To a 10-ml aliquot add 2% Na_3AsO_3 soln. (10 ml), heat to boiling, cool, add aq. NH_3 - NH_4Cl buffer soln. (25 ml) at pH 9 and 0·5% gelatin soln. (5 ml), make up to 100 ml and proceed as before. The relative error in the determination of technical products (**I** or **II**) is about ± 2%. The polarographic characteristics of **I** and **II** are examined; in the case of **II** they are not affected by treatment with Na_3AsO_3. The results show that the determination of **II** in the presence of **I** is feasible. This polarographic method is of particular importance in following the nitrification process in the synthesis of **II**. K. R. C.

3054. **Ultra-violet and infra-red spectra of 1:2:3-triazole derivatives.** E. Lieber, C. N. R. Rao, T. S. Chao and H. Rubinstein (Dept. of Chem., De Paul Univ., Chicago, Ill., U.S.A.). *Canad. J. Chem.*, 1958, **36** (10), 1441–1443.—The spectra recorded are of 1:2:3-triazoles having R_1 (the substituent in the 1-position) = C_6H_5, H, *p*-BrC_6H_4 or $CH_2C_6H_5$, R_4 (the substituent in the 3-position) = H, COOH, $COOCH_3$ or C_6H_5, and R_5 (the substituent in the 2-position) = OH, Cl, NH_2, NHC_6H_5 or $NHC_6H_4.CH_3(p)$. Absorption in the near u.v. is independent of R_1, R_4 and R_5. All derivatives show a band of weak or medium intensity at 1277 to 1302 cm^{-1}, whilst the typical bands of the triazole ring appear at 1003, 1101 to 1129, and 1045 to 1093 cm^{-1}. The spectra are discussed in respect of mol. structure and the frequency assignments reported previously (Hartzel and Benson, *J. Amer. Chem. Soc.*, 1954, **76**, 667; Lieber *et al.*, *Spectrochim. Acta*, 1950, **10**, 250). W. J. BAKER

3055. **Nuclear magnetic resonance in petroleum analytical research.** R. L. Williams (Humble Oil and Refining Co., Baytown, Texas, U.S.A.). *Spectrochim. Acta*, 1959, **14**, 24–44.—About 500 spectra from high-resolution measurements of proton resonances in free-flowing liquid samples free from appreciable paramagnetic impurities have been collected. Analysis of complex spectra is discussed, but it is pointed out that the solution is obvious in 50% of the cases. It is considered more likely that the method will be of value for functional group determination and structural work on isomers than for the determination of whole structures. In an example, four types of H are shown to give bands of equal width, in which case absorption can be determined very accurately from peak height to give answers as weight per cent. at the rate of 20 min. per assay. Statistics given show no large systematic errors for such a method. An investigation of the relation between rate of proton exchange and band broadening or band merging phenomena is reported. N.M.R. characterisation schemes for aromatic and saturate concentrates separated from high-boiling petroleum fractions by chromatographic methods are briefly described and have been discussed more fully elsewhere (*cf.*

ASTM Publ. S.T.P. 224). The direct absorption method for the determination of total hydrogen in liquids of high hydrogen content is given in detail, and the more sensitive absorption mode and dispersion mode modulation methods are discussed. Typical results for the direct method show a coeff. of variation of ± 2%. Greater precision in certain directions is possible. E. G. CUMMINS

3056. **Structural group analysis of mineral-oil fractions. II. Structural group analysis by infra-red spectroscopy.** G. Brandes (Dtsch. Erdöl A.-G., Hamburg). *Erdöl u. Kohle*, 1958, **11** (10), 700–702.—In the mol.-wt. range 290 to 500 all the relevant aromatic systems show an equally strong extinction at 1610 cm^{-1}. Below 290, the range over which any particular expression is valid becomes increasingly narrow. The expression, aromatic content, $C_A = 1{\cdot}2 + 9{\cdot}8E^{1610\,cm^{-1}}_{max.}$ is valid in the concn. range $C_A = 0$ to 60%. A correction for the effect of C=O bands is made by multiplying the extinction at 1610 cm^{-1} by $(1 + 0{\cdot}15E^{C=O}_{max.})$. The max. extinction for C=O lies between 1700 and 1750 cm^{-1}. The paraffin content, C_P, is determined from the absorption bands at 720 cm^{-1} by means of the expression $C_P = 29{\cdot}9 + 6{\cdot}6E^{720\,cm^{-1}}_{max.}$. This expression gives a low value for paraffins if the aromatic content is very small, and a high value if the aromatic content is high. In the range of about 8 to 25% of aromatics this effect gives an error of < 1·5%. Below 8% of aromatics a correction can be made for the aromatic content; if the aromatic content exceeds 25%, the paraffin bands cannot be evaluated. For oils with a high pour-point, the extinction may be measured at between 60° and 70°, a correction being made for temp. The extinction at 25° is 1·033 times that at 65°. The reproducibility is ± 3·5%, relative, for C_A and ± 2% for C_P. The naphthene content can thus be obtained with a reproducibility of ±1·5% (absolute).

III. Applicability of structural group analysis to the evaluation of lubricating oil distillates and raffinates. G. Brandes. *Ibid.*, 1958, **11** (11), 781–785.—Structural group analysis enables oils to be classified quant. into paraffin-based, naphthene-based or mixed base. The structural composition of more than 100 oils has been determined by i.r. spectroscopy, which is quicker and more precise than the *n-d-*M method. For the i.r. method see Brandes (*Anal. Abstr.*, 1957, **4**, 4032; and Part II, above). The utility of the method is discussed in relation to the source of the oil, its refining characteristics and its temp. - viscosity characteristics. The max. viscosity index and the yield attainable from a given oil by solvent refining can be deduced.

G. BURGER

3057. **Exhaust gas analysis by gas chromatography combined with infra-red detection.** W. B. Heaton and J. T. Wentworth (Res. Lab., General Motors Corp., Detroit, Mich., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 349–357.—In the method described, the eluate from the gas-chromatographic column is burned to convert the hydrocarbons into CO_2 and H_2O. It is then passed through a non-dispersive i.r. analyser, sensitised to CO_2. Peaks can be obtained with hydrocarbon concn. as low as 1 p.p.m. K. A. PROCTOR

3058. **Analysis of polycyclic aromatic hydrocarbons in the exhaust gases of Diesel engines.** A. Reuter, K. O. Johne and J. Lieb (Battelle Inst., Frankfurt am Main, Germany). *Z. anal. Chem.*, 1958, **164** (3), 335–337.—Anthracene, pyrene, 3:4-benzopyrene, 1:12-benzoperylene, fluoranthene and 11:12-benzofluoranthene have been found in Diesel soot. The exhaust gases are passed via a pilot tube and pressure impact release valve over an asbestos filter. The soot is collected on the filter from 500 litres of the exhaust gases at 50 to 100 litres per min. The filter and soot are broken up and extracted with benzene. After filtration, the solvent is evaporated, the residue is dissolved in *cyclo*hexane, passed through an Al_2O_3 column (15 cm × 0·7 cm) and developed with *cyclo*hexane - diethyl ether (9:1) until the 5th fraction has left the column. Each fraction is further separated by one-, and in individual cases two-, dimensional paper chromatography. Acetylated (20 to 25%) paper is used and separation is effected by the use of the solvent systems methanol - diethyl ether - water (4:4:1) or methanol - $CHCl_3$ (3:10). The spots which fluoresce in u.v. light are eluted with diethyl ether or methanol, and measured by u.v. spectroscopy.

B. B. BAUMINGER

3059. **Improvements in or relating to methods and devices for testing for the presence of water in suspension in other liquids.** "Shell" Research, Ltd. [Inventors: J. R. Joyce, K. R. Williams and D. Anson]. Brit. Pat. 806,928; date appl. 22.3.56.—The liquid (aviation kerosine) is caused to flow through a disc of filter-paper coated with a powder containing a highly coloured dye which is sol. in water, but not in the other liquid, *e.g.*, crystal violet, fluorescein, magenta, Rhodamine B 500 or a mixture of inorganic salts which react in aq. soln. to give a coloured product. J. M. JACOBS

3060. **New method of analysis of petroleum oil and bitumen without preliminary ashing.** V. I. Malinina and V. P. Varlamov (All-Union Sci. Res. Geological Prospecting Petroleum Inst.). *Zavod. Lab.*, 1958, **24** (11), 1374–1375.—To determine traces of Mn, Ni, V, Fe, Cu, Na and Ti in oil and bitumen, the sample (150 to 250 g) is heated at 300° to remove volatile matter and to give a coke which is then powdered. The arc spectrum (8 to 12 amp.) of a portion (100 mg) on a strip of cigarette paper (350 mm × 20 mm) which has been moistened with 30% $(NH_4)_2SO_4$ soln. and dried is compared with standards. G. S. SMITH

3061. **Determination of sodium in residual fuel oil using a.c. spark and rotating platform electrode.** R. S. Hullings (Westinghouse Electric Corp., Lester, Pa., U.S.A.). *Appl. Spectroscopy*, 1958, **12** (5), 139–140.—The analysis is performed directly on the oil, with the rotating platform electrode technique. Sodium in the range from 2 to 100 p.p.m. can be determined with an accuracy of ≃10% at the higher concn. of 50 to 100 p.p.m. and ≃30% in the range from 5 to 20 p.p.m. In the higher concn. range the standard deviation is ≃ ±2 p.p.m. and in the lower ≃ ± 1·5 p.p.m. Better reproducibility is obtained with the rotating electrode as the lower electrode rather than the reverse. The method may also be adapted for the determination of Ca, Mg, V, Ni and Al.

K. A. PROCTOR

3062. **Analysis of hydrocarbon waxes by fluorescence spectroscopy.** H.-J. Eichhoff and G. Titschack (Univ., Mainz). *Erdöl u. Kohle*, 1958, **11** (10), 705–708.—Because of their carcinogenic properties,

the permissible concn. of certain aromatic polycyclic hydrocarbons in paraffin waxes for food packing is very small. The compounds include methylcholanthrene, anthracene, dibenzanthracene and 3:4-benzopyrene, which can be detected and determined in sufficiently small concn. by fluorescence spectroscopy. The instrument used is fitted with a high-pressure xenon lamp and an exit slit and photomultiplier which traverse the focal plane. Filtered u.v. light is focused on to the specimen, and the secondary radiation at 45° is directed by a mirror into the slit of the spectrometer. A high resolving power is necessary. No correction is needed for the spectral sensitivity of the photocell. For an unknown sample, the spectrum must be compared with that of the same paraffin with contaminants added; fluorescence under a u.v. lamp is not proof of their presence. The manufacture of a wax can be followed by fluorescence spectroscopy. The starting material may show no bands because of the presence of fluorescence quenchers. The bands may then appear during refining and disappear again as the polycyclic aromatics are removed. Normal refining methods yield wax free from these impurities.

G. BURGER

3063. **Determination of carbon and hydrogen in coal and coke by the Liebig method. Elimination of errors due to formation of nitrogen dioxide.** M. P. Mendoza (Brit. Coal Utilis. Res. Ass., Randalls Rd., Leatherhead, England). *Fuel, Lond.*, 1958, **37** (4), 409–414.—The silver gauze in the combustion tube packed according to the B.S. 1016: 1942 method does not retain all oxides of N formed during the combustion of coal, and these cause high values for C (≃ 0·2%) and H (≃ 0·05%). The NO_2 is absorbed by granular MnO_2 (*cf.* Belcher and Ingram, *Brit. Abstr. C*, 1950, 386; 1951, 33), but since this reagent also absorbs H_2O vapour it can be used only to eliminate the error in the carbon value, not that for H. A. R. PEARSON

3064. **Evolution of oxides of nitrogen and of sulphur during the determination of carbon and hydrogen in coal.** A. H. Edwards (Nat. Coal. Bd., Coal Survey Lab., Newcastle upon Tyne). *Fuel, Lond.*, 1958, **37** (4), 415–420.—Oxides of S are completely arrested by $PbCrO_4$ in the standard combustion-tube packing (B.S. 1016:1942). Oxides of N may cause errors which average + 0·17% for C and − 0·02% for H, the latter being considered insignificant. The NO_2 is completely arrested by MnO_2 (*cf. Brit. Abstr. C*, 1950, 386, 440). In the Sheffield high-temp. combustion, only negligible amounts of NO_2 are produced (*cf.* Mott and Wilkinson, *Anal. Abstr.*, 1956, **3**, 2464).

A. R. PEARSON

3065. **Determination of humic acid in local and imported coals.** Ž. Panković and N. Gvozdarević (Inst. za Ugal, Belgrade). *Bull. Soc. Chim. Belgrade*, 1956, **21** (6–10), 477–480.—The results are tabulated of humic acid determinations on twelve Yugoslav and six imported coals. Gravimetric determinations by extraction with NaOH or Na_2CO_3 followed by pptn. with dil. HCl, determinations with $KMnO_4$ and the Ca acetate method were used. Results indicated that none of these methods was sufficiently reliable and considerable discrepancies were shown. A. GROCHOWSKI

3066. **The analysis of synthetic detergents. A review.** W. B. Smith (Marchon Products Ltd., Whitehaven, Cumberland, England). *Analyst*, 1959, **84**, 77–89.—The review deals mainly with the domestic spray-dried powder detergent. The surfactants constituting these are classified and qualitative tests are described. The quantitative methods reviewed include extraction methods, colorimetric determinations, anionic - cationic titrations, ion-exchange methods and precipitation methods. Finally methods for the determination of the inorganic constituents of detergents are reviewed. (80 references.) A. O. JONES

3067. **Essential oils.** British Standards Inst. (2 Park St., London). B.S. 2999/16 to 31, 1959, 37 pp.—This second volume includes specifications for oils of bay; cassia; Ceylon citronella; Java citronella; clove bud; clove leaf; clove stem; *Eucalyptus citriodora*; geranium, Kenya; geranium, N. Africa; geranium, Réunion (Bourbon); petitgrain, Paraguay; pimento berry; pimento leaf; E. Indian sandalwood and W. Australian sandalwood. H. B. HEATH

3068. **Application of gas chromatography to the analysis of synthetic and natural aromatic substances.** E. Bayer, G. Kupfer and K.-H. Reuther (Forsch.-Inst. f. Rebenzüchtung, Abt. Biochem. u. Physiol., Geilweilerhof über Landau, Pfalz, Germany). *Z. anal. Chem.*, 1958, **164** (1), 1–10.—The gas-chromatographic separation of essential oils containing terpenes, with special reference to geranium oil, is described. Retention vol. on different stationary phases at various temp. are recorded. It is shown that gas chromatography, in conjunction with other physical and chemical methods, can be used for resolving the composition of essential oils. Eight components were identified in geranium oil. B. B. BAUMINGER

3069. **Specific quantitative colorimetric method of analysis for citral in lemon oil.** W. L. Stanley, R. C. Lindwall and S. H. Vannier (Fruit and Vegetable Lab., Western Utilization Res. and Devel. Div., Agric. Res. Service, U.S. Dept. of Agric., Pasadena, Calif.). *J. Agric. Food Chem.*, 1958, **6** (11), 858–860.—Previous methods for determining citral in lemon oil were non-specific, and measured only total carbonyl content. The specific method described involves reaction with a mixture of vanillin and piperidine in absolute methanol or ethanol, to form an alcohol-soluble emerald-green complex, with max. absorption at 605 mμ. Only dihydrocitral and *pseudo*ionone interfere; other carbonyl compounds give yellow, orange or red colours. M. D. ANDERSON

3070. **Spectrophotometric investigation of essential oils. II. Evaluation of lemongrass oil through ultra-violet spectrophotometry.** K. L. Surve, K. K. Chakravarti and S. C. Bhattacharyya (Nat. Chem. Lab., Poona, India). *Perfum. Essent. Oil Rec.*, 1958, **49** (10), 624–626.—A method for the determination of the citral content of East Indian lemongrass oil, based on the optical density of an ethanolic soln. at 238 mμ ($\epsilon = 14{,}787$), is described. Results by this method are ≃ 5% lower than those obtained by the hydroxylamine hydrochloride or bisulphite methods. The presence of myrcene may cause interference in the evaluation of West Indian lemongrass oil. Geraniol does not interfere with the determination. H. B. HEATH

3071. **Determination of cineole in essential oils of eucalyptus and niaouli by gas - liquid partition chromatography.** L. Domange and S. Longuevalle (Lab. Nat. de Santé Publ., Sect. des Médicaments, France). *Ann. Pharm. Franç.*, 1958, **16** (9–10), 557–561.—The oils are chromatographed on silicone E.301 (30% on Celite) at 160° with N as carrier gas on a column 1·7 metres × 8 mm, and with a flow rate of 1 litre per min. between pressures of 360 and 145 mm. The oil to be examined is introduced with *cyclo*hexanol as internal standard with a retention time of 3 min. while cineole has a retention time of 6 min. Chloroform at this temp. passes directly through the column. The calibration curve is established with known mixtures by plotting the ratio of the peaks of the two compounds against the ratio of their weights. The plot is linear. The cineole content of 3 eucalyptus and 3 niaouli oils is investigated both directly and by adding a known quantity of cineole and the method is considered superior to the *o*-cresol method of the French Codex.
E. J. H. BIRCH

3072. **Analysis of mixed cellulose esters.** E. Schröder, J. Franz and K. Thinius (Inst. Chem. Tech. Plaste, Leipzig, E. Germany). *Plaste u. Kautsch.*, 1958, **5** (11), 411–415.—Cellulose acetate butyrate is saponified for 15 hr. at 40° with alcoholic NaOH, the cellulose is filtered off, the alcohol removed by distillation, and, after passage of an aq. soln. of the residue through an ion-exchange column (H form), the eluate can be titrated with alkali and the sap. val. obtained. The acetic and butyric acids are separated by partition chromatography on a silica-gel column, with H_2O as the stationary phase and $CHCl_3$ - *n*-butanol as the mobile phase, the various fractions being titrated with 0·02 *N* NaOH, with phenol red as indicator. Application of the procedure to the full analysis of cellulose acetate butyrates is described, satisfactory weight balances being obtained. L. A. O'NEILL

3073. **Determination of dialkyltin compounds in poly(vinyl chloride).** A. H. Chapman, M. W. Duckworth and J. W. Price (Tin Res. Inst., Perivale, Mddx., England). *Brit. Plast.*, 1958, **32** (2), 78.—Dialkyltin compounds are determined by measuring the extinction of a complex formed with dithizone in the presence of an EDTA - borate buffer at pH 8. Any other metals which may be present are removed by extraction with HCl. A sample of poly(vinyl chloride) containing < 700 μg of Sn is heated under reflux in a 150-ml flask with 25 ml of 1:2-dichloroethane until dissolved. Then 25 ml of HCl is added and heating is continued for a further 5 min. The contents of the flask are transferred to a separating-funnel, the solvent layer is removed and the HCl layer washed twice. The solvent layer and washings are made up to 50 ml. An aliquot of the soln., 5 ml of buffer soln. [19 g of $Na_2B_4O_7.10H_2O$, 12 g of H_3BO_3 and 2·5 g of EDTA (disodium salt) made up to 1 litre with water] and 1 ml of dithizone soln. are shaken in a separating-funnel, the dithizone layer is separated and its extinction measured at 450 mμ and compared with a standard. The accuracy is ≃ ±2%. S.C.I. ABSTR.

3074. **Determination of chlorinated rubber and poly(vinyl chloride) in the presence of each other.** G. Norwitz (Pitman-Dunn Lab., Philadelphia). *Off. Dig. Fed. Paint Varn. Prod. Cl.*, 1958, **30**, 1049–1058.—Published work on the analytical chemistry of chlorinated rubber (**I**) and poly(vinyl chloride) (**II**) was considered in developing a procedure for the determination of these substances in the presence of each other. A method is proposed whereby **I** is separated from **II** by the use of $CHCl_3$, which dissolves **I** but not **II**. The chlorine content of **I** and **II** is then determined by a procedure in which the sample is mixed with Na_2CO_3 in a large platinum crucible, a smaller crucible is inserted upside down into the large one, and the space between the two is packed with MgO. On heating, the Na_2CO_3 melts but the MgO does not. The Na_2CO_3 and MgO are dissolved in HNO_3 and the Cl determined gravimetrically as AgCl. D. R. DUNCAN

3075. **Determination of organically combined metals in poly(vinyl chloride).** E. Schröder and S. Malz (Inst. Chem. Tech. Plaste, Leipzig, E. Germany). *Plaste u. Kautsch.*, 1958, **5** (11), 416–417.—The poly(vinyl chloride), *e.g.*, as foil, is subjected to wet oxidation with H_2SO_4 - HNO_3, and a qual. analysis for metals is carried out on the residue by chemical or other methods; quant. analysis is carried out polarographically on soln. of the residue, the procedures to be followed for Cd, Pb and Sn being described. L. A. O'NEILL

3076. **The use of derivative spectroscopy for determining methyl groups in polythene.** G. L. Collier and A. C. M. Panting (I.C.I. Ltd., Alkali Div., Northwich, Ches., England). *Spectrochim. Acta*, 1959, **14**, 104–118.—Difficulties due to the overlap of the methylene and methyl absorption bands at 1365 and 1378 cm^{-1}, respectively, have been overcome by the method described, so that < 2 methyl groups per 100 carbon atoms can be accurately determined. Overlap in the second derivative spectrum can be detected by observation of the nature of the calibration diagram, as illustrated. The principles are stated and used to show that in the second derivative there is no overlap between the 1378 cm^{-1} methyl band and adjacent methylene absorptions when measurements are made at 1378 cm^{-1}. The circuit diagram of the logarithmic circuit is appended. Alternating current from the spectrometer is converted to direct current by a synchronous rectifier and integrator type network to provide distortion-free rectification down to low-signal level. The complete characteristic can be checked in 3 to 4 min. and new conditions after valve replacement can be confirmed in 30 min. E. G. CUMMINS

3077. **Rapid identification of dicarboxylic acids in alkyd resins and polyesters.** G. G. Esposito and M. H. Swann (U.S. Army Ordnance, Aberdeen Proving Ground, Md., U.S.A.). *Off. Dig. Fed. Paint Varn. Prod. Cl.*, 1958, **30**, 1059–1069.—The alkyd resin or polyester is saponified with KOH in anhydrous ethanol in the presence of excess of benzene. The pptd. dipotassium salts of the dicarboxylic acids are filtered off, washed and dried. Weighed amounts of the salts are then treated with H_2O and various amounts of 6 *N* HCl or acetic acid in a series of 11 operations. When crystallisation of acid occurs the crystals are examined microscopically. The acids are identified according to the conditions under which they crystallise and the appearance of the crystals. The method is mainly limited to single acids.
L. A. O'NEILL

3078. **Rapid determination of the metal content of paint driers.** Chicago Paint & Varnish Production Club. *Off. Dig. Fed. Paint Varn. Prod. Cl.*, 1958, **30**, 1178–1187.—The drier (0·2 g) is dissolved in ethanol - benzene (25:1), a known excess of 0·01 *M* EDTA (disodium salt) is added and the excess is back-titrated with 0·01 *M* $ZnCl_2$, with Eriochrome black T as indicator, in the presence of an ammoniacal NH_4Cl buffer. The method is applicable to Ca, Zn and Co, but with the last-named the back-titration must be completed rapidly. For Pb, tartaric acid, and for Mn, ascorbic acid, are added before the buffer soln. Results compare favourably with those by gravimetry and flame photometry, and a determination requires only $\simeq$ 10 min. L. A. O'NEILL

3079. **Identification of lacquers and adhesives on wood shavings.** K. Seifert (Inst. für Holztechnol., Dresden). *Plaste u. Kautsch.*, 1957, **4** (11), 445–447.—A scheme for the identification of adhesives and coatings on small shavings of wood, *e.g.*, from furniture, is outlined. Nitrocellulose, phenolic and urea resins, poly(vinyl acetate), casein, glue and starch are recognised by treatments with bromophenol blue, phenolphthalein, bromothymol blue, resorcinol - HCl and I - KI by observing microscopically the colours produced on the wood and on the organic film by transmitted and reflected light. L. A. O'NEILL

3080. **Carnauba wax analysis.** C. S. Treacy and A. S. Cascione (M. Argueso & Co., Mamaroneck, N.Y.). *Soap Chem. Specialties*, 1958, **34** (10), 97, 103–104.—Hydrocarbon determination is accelerated and resin extraction made more accurate by the use of a novel extraction tube (described). The tube is charged with dry, activated Al_2O_3, the apparatus being set up as in A.S.T.M. D–1342–54T, and boiling heptane is run in. Wax (4 g) dissolved in heptane (75 ml) is poured slowly into the top of the tube. The extract is heated under reflux, the tube being between the flask and the condenser. The heptane from the flask is then evaporated, and the hydrocarbons are weighed. For determination of resin, finely ground carnauba is leached at 15° with acetone, and the residue is weighed after the wax has been filtered off and the acetone evaporated. S.C.I. ABSTR.

3081. **Methods of testing rubber latex.** British Standards Institution (2 Park Street, London). B.S. 1672: Part 2:1954. Amendment No. 1, published 16.3.59, 8 pp.—The method for the determination of Cu has been replaced by two methods, one of which depends on extraction of a soln. of the ash with a $CHCl_3$ soln. of Zn diethyldithiocarbamate after complexing the Fe with ammonium citrate, and measuring the colour of the extract; in the other method, Na diethyldithiocarbamate is added and the colour is extracted with CCl_4. A method for the determination of total Mn depends on oxidation with KIO_4, and a complexometric method is described for Mg.

3082. **Zirconium tannage. I. Colorimetric method for the determination of zirconium in leather.** D. A. Williams-Wynn (Leath. Ind. Res. Inst., Rhodes Univ., Grahamstown, S. Africa). *J. Soc. Leath. Tr. Chem.*, 1958, **42** (11), 360–368.—*Procedure*—The leather (0·3 g containing 10% of ZrO_2) is digested with $HClO_4$ - H_2SO_4 (2:1) (10 to 15 ml) and conc. HNO_3 (15 ml), and the digest is cooled, diluted with *N* HCl (50 ml), boiled for 5 min. and diluted with H_2O to 500 ml. An aliquot (10 ml) is added to *N* HCl (25 ml) in a 100-ml flask. Alizarin red S (0·1%) (20 ml) and Na tartrate (0·5%) (20 ml) are added. The soln. is heated in boiling water for 5 min., then cooled and made up to 100 ml, and the extinction is measured at 520 mμ. Titanium, Al, Fe and Cr^{3+} do not interfere; Cr^{6+} must be reduced before carrying out the determination. C. A. SLATER

3083. **Chemical examination of ink script.** K. Koopmans (Gerechtelijk Lab., Min. van Justitie, 's-Gravenhage, Holland). *Chem. Weekbl.*, 1959, **55** (11), 109–114.—Iron and sulphate can be semi-quant. determined by microphotometric examination of as little as 2 to 5 mm of an ink line. The paper is rendered transparent with benzaldehyde, and the extinction of the script is measured at a series of points along a cross-sectional line, and plotted with respect to position. The area bounded by the curve and the enclosing horizontal representing complete transmission is measured as the *S*-value. After removal of the benzaldehyde, the strip is treated with aq. acid $K_4Fe(CN)_6$, washed, and then treated with aq. acid $KMnO_4$ to destroy the organic colouring matter. After treatment with Na_2SO_3 soln., washing, and drying, the *S*-value of the Fe (as Prussian blue) is determined on the lines previously described by extinction measurements made at the original points. The *S*-value of the sulphate is determined by fixation as $PbSO_4$ and conversion of the $PbSO_4$ into PbS. The *S*-values thus determined (especially those for Fe) often serve as means for distinction between different makes of ink. Some inks which have the same *S*-values may produce different chromatograms, and *vice versa*. P. S. ARUP

3084. **Infra-red quantitative analysis data.** *Anal. Chem.*, 1959, **31** (5), 959–960.—The following data are published. **Analysis of detergent-slurry components,** V. A. Cirillo. **Determination of poly(vinyl cyanide) in coatings,** M. L. Adams and M. H. Swann. **Determination of naphthalene and ethyl chloride in tetraethyl-lead,** R. P. Curry. **Determination of acetate and formate in geranium Algerian and geranium Bourbon [oils],** A. J. Fenton. **Determination of hex-1-yne and hex-1-ene in *iso*-octane solution,** R. P. Curry.

See also Abstracts—**2825**, Essential oils and related products (review). Solid and gaseous fuels (review). Petroleum (review). Natural and synthetic rubbers (review). **2827**, Physical methods in analysis of olefins. **2843**, Use of i.r. spectrophotometry in organic analysis. **2855**, Determination of water in paper. **2913**, Determination of Cu in rubber chemicals and products. **2887**, Determination of ZnO in pigments. **2909**, Determination of Pb in materials for protective coatings. **2910**, Determination of Pb in organic materials. **2922**, Determination of hydrazine deriv. **2923**, Determination of nitroethane. **2936**, Determination of sulphonic acids. **3175**, Determination of methylal in formalin. **3181**, Determination of amylose in starch. **3198**, Analysis of acid casein. **3202**, **3203**, **3204**, **3205**, **3206**, Separation of dyes used in food. **3234**, Determination of acetylene in air. **3235** Analysis of solvents injurious to health. **3277** Detection of water in halogenated refrigerants **3303**, Gas chromatography of aromatic hydrocarbons.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Biological fluids, animal and vegetable tissues

3085. **Neutron activation analysis of blood.** R. P. Spencer, T. G. Mitchell and E. R. King (Radioisotope Lab., U.S. Naval Hosp., Bethesda, Md.). *Amer. J. Roentgenol.*, 1958, **79**, 1053–1062.—Neutron activation analysis consists in bombarding the element or elements concerned with neutrons and measuring the radioactivity thus induced. Only very small amounts of material are needed and the method is not destructive. The calculations in this article are based on a neutron flux of $2{\cdot}5 \times 10^8$ neutrons per sq. cm per sec., obtainable from a laboratory source of neutrons such as a Ra - Be mixture or a Be source in conjunction with a Van de Graaff high-voltage generator. If 10 ml of plasma is exposed to this flux for 1 hr., isotopes of Na, Mg, K, Cu, Zn, Ca, Cl, I, Br, P, Co and Mn are found. By far the greatest amount of γ-radiation emitted from the activated blood comes from ^{24}Na and ^{38}Cl, which are the only nuclides detectable directly. The composition of erythrocytes may be determined directly by activation analysis because they have low concn. of Na and Cl, but these substances have to be removed from whole blood before the other elements can be determined. If Na and Cl are removed before irradiation, ions must be used that do not themselves become radioactive; NH_4^+, NO_3^-, CN^- and certain org. radicals are suitable. If Na and Cl are removed after irradiation a rapid method must be used, otherwise radioactive decay becomes important. NUTR. ABSTR. REV.

3086. **Determination of the hepatic blood flow by means of ethanol. Preliminary report.** J. A. Larsen (Dept. of Surgery, Finsen Inst., Copenhagen, Denmark). *Scand. J. Clin. Lab. Invest.*, 1958, **10** (3), 263–265.—The determination of the concn. of blood ethanol at regular intervals during an intravenous injection of ethanol in isotonic saline soln. (60 g of ethanol per litre), and a plot of the results against time, enables hepatic blood flow to be determined. The method is simple and overcomes the disadvantages of hepatic catheterisation. Even though ethanol is known to increase hepatic blood flow this method should still be useful for the diagnosis of impaired function. Galactose can be used instead of ethanol, and this method is being developed. D. B. PALMER

3087. **Bubble method for determination of pCO_2 and pO_2 in [arterial] blood.** E. Asmussen and M. Nielsen (Lab. for the Theory of Gymnastics, Univ., Copenhagen, Denmark). *Scand. J. Clin. Lab. Invest.*, 1958, **10** (3), 267–271.—The procedure is based on the bubble method of Krogh (1908) as modified by various authors. A bubble of approx. 5 cu. mm is equilibrated in 3 ml of blood at 37° for 10 to 15 min., and is then transferred to a micro-gas analyser (described). The method was tested with air-bubbles of various compositions and the results showed errors of $+1 \pm 2$ mm of Hg for pCO_2 and -1 ± 2 mm of Hg for pO_2. D. B. PALMER

3088. **Icterus index determined with the spectrophotometer corrected for turbidity and haemoglobin.** J. Fog (Med. Dept., Univ. Clinic, Rikshosp., Oslo, Norway). *Scand. J. Clin. Lab. Invest.*, 1958, **10** (3), 246–249.—The icterus index is measured by determining the maximum absorption of serum at 460 mμ. A correction, necessary for accurate results, and its theoretical aspects are discussed and the construction of a nomogram is described. The values obtained by this method have a coeff. of variation of 1·9 to 3·2%. D. B. PALMER

3089. **The distribution of "fall-out" radiostrontium in a sheep skeleton. [Determination of strontium-89 and strontium-90 in animal bone.]** A. Morgan and J. E. Wilkins (Health Physics Division, A.E.R.E., Harwell, Berks.). *Biochem. J.*, 1959, **71** (3), 419–422.—The determination is based on that described by Bryant *et al.* (Unclassified A.E.R.E. Rep., 1956, HP/R 2056). After Sr carrier has been added to the ashed bone, successive separations with fuming HNO_3 are used to remove Ca. Possible Ra contamination is removed by scavenging ppt. of $BaCrO_4$ and $Fe(OH)_3$. The purified Sr is stored with Y carrier while the ^{90}Y equilibrates with the ^{90}Sr. Finally the Y is separated and the ^{90}Y radioactivity is counted. From its activity, that of the ^{90}Sr is calculated. The Sr is counted after the ^{90}Y has re-equilibrated and the ^{90}Sr - ^{90}Y contribution is deducted to give the ^{89}Sr. J. N. ASHLEY

3090. **Detection of yellow phosphorus and phosphides in biological material.** A. S. Curry, E. R. Rutter and Lim Chin-Hua (Home Office Forensic Sci. Lab., Harrogate, England). *J. Pharm. Pharmacol.*, 1958, **10** (10), 635–637.—The micro-test of Schwartz *et al.* (*cf. Anal. Abstr.*, 1956, **3**, 3417) has been modified to increase the specificity. Instead of air, CO_2 is used as carrier gas to distil the P on to the $AgNO_3$-impregnated filter-paper. The silver phosphide stain is oxidised with Cl gas instead of with hypochlorite. The PO_4^{3-} are separated from AsO_4^{3-} and Ag^+ on the filter-paper by electrophoresis for 3 hr. in 5% aq. acetic acid as electrolyte. The paper is dried and the blue colour of the molybdophosphate developed by the method of Hanes and Isherwood (*Nature*, 1949, **164**, 1107). A. R. ROGERS

3091. **Simultaneous determination of chromium-51 and iodine-131 activities in doubly labelled blood.** R. Adams, I. C. Woodward, M. G. Crane and J. E. Holloway. *J. Lab. Clin. Med.*, 1958, **52** (5), 754–761.—For the measurement of comparative flow rates of red blood cells and plasma in continuous blood-sample analysis, cells labelled with ^{51}Cr and plasma labelled with ^{131}I are employed, and the γ-activities of the two isotopes (0·32 and 0·364 MeV, respectively) are measured simultaneously in a two-channel pulse-height analyser. The result for each isotope is calculated from the simultaneous equations given. Results for ^{51}Cr averaged $-1{\cdot}35\%$ (coeff. of variation $\pm$ 2·26%) and for ^{131}I, $+0{\cdot}11\%$ (coeff. of variation $\pm$ 2·39%) on mixed samples prepared gravimetrically. The method is of general application to pairs of γ-emitting isotopes with sufficient difference in γ-energies for separate counting. W. H. C. SHAW

3092. **Determination of fluoride in blood serum.** L. Singer and W. D. Armstrong (Dept. of Physiol. Chem., Univ. of Minnesota, Minneapolis). *Anal. Chem.*, 1959, **31** (1), 105–109.—Serum or plasma (5 to 10 ml) is ashed in a platinum dish with MgO (of low fluoride content) as a fluoride-fixative, heating finally at 500° for 75 min. The ash is

transferred to a micro-distillation apparatus with two 1-ml portions of cold 30% $HClO_4$ and 2 ml of 60% acid. The distillation tube is heated in a glycerol bath held at 145° to 150° and a stream of nitrogen is used to sweep the fluoride into the receiver, thus reducing the volume of distillate obtained with the usual steam-distillation; 15 ml of H_2O is added dropwise during distillation. Fluoride is determined spectrophotometrically in the distillate at 568 mμ by the fading caused by fluoride of a Zr - Eriochrome cyanine R lake (1 vol. of Eriochrome cyanine R soln. containing 2·1067 g per litre and 2 vol. of zirconyl chloride soln. containing 0·3046 g in 1614 ml of conc. HCl diluted to 2 litres with H_2O, freshly mixed) and comparison with fluoride standards. The mean recovery of 2 μg of fluoride as NaF was 101 ± 3·1% (32 determinations), and for 3 or 4 μg the recovery was 100·8 ± 3·7% (40 determinations). The colorimetric method used is compensated for chloride and is applicable up to 3 μg of fluoride.

D. W. Moss

3093. **Determination of the concentration of 4-aminosalicylic acid in blood, tissue, juices and secretions by means of ferric chloride and diazotised sulphanilic acid.** V. A. Temmere and K. A. Seglin'sh. *Trudÿ Inst. Exsp. Med. Akad. Nauk Latv. SSR*, **15**, 57–68; *Ref. Zhur., Khim., Biol. Khim.*, 1959, (6), Abstr. No. 6369.—The first method described is based on the ability of 4-aminosalicylic acid (**I**) to form at pH ≃ 2 a red-violet complex with $FeCl_3$. The colour is determined colorimetrically. In the second method, $NaNO_2$ is added to a soln. of sulphanilic acid in HCl. The diazotised sulphanilic acid forms a yellow-orange compound with **I** which is determined colorimetrically. Both methods are used to determine free and acetylated **I**.

K. R. C.

3094. **Rapid quantitative method for chemical determination of urinary catecholamines in the diagnosis of phaeochromocytoma.** J. T. Wright (London Hosp., London). *Lancet*, 1958, **ii**, 1155–1157.—Acidified urine is passed through a column (approx. 8 cm) of Dowex 2 (acetate) to remove mineral and strong organic acids. Boric acid soln. is added to the percolate and the soln. is passed through a second column of Dowex 2 (borate). The catecholamines are eluted from the latter with 0·2 *N* HCl, and noradrenaline and adrenaline are determined fluorimetrically.

H. F. W. Kirkpatrick

3095. **Determination of 1-(5-nitro-2-furfurylideneamino)hydantoin [nitrofurantoin] in urine.** B. Zaar (Dept. Clin. Chem., Univ. Hosp., Uppsala, Sweden). *Scand. J. Clin. Lab. Invest.*, 1958, **10** (4), 432–434.—Urine is diluted with a phosphate-borate buffer and is shaken with a 1% soln. of benzethonium chloride in $CHCl_3$. A yellow complex is formed which dissolves in the $CHCl_3$ layer. After separation and filtration, the extinction coefficient of the soln. is determined photometrically at 400 mμ, before and after the addition of glacial acetic acid. The difference between the two readings is proportional to the amount of nitrofurantoin present. Quant. extraction is achieved within the range 0 to 20 mg per 100 ml. The complex is light-sensitive.

D. B. Palmer

3096. **Identification and determination of diethyl *p*-nitrophenyl phosphorothionate [parathion] in tissue and blood.** E. Vidic (Inst. Forensic and Social Med., Free Univ., Berlin). *Arzneimittel-Forsch.*, 1958, **8** (11), 719–726.—Parathion (**I**) in tissue is recovered by extraction with methanol and purified by treatment with activated charcoal and chromatography in dichloromethane soln. on specially activated alumina. Recovery from blood is carried out by extraction with dichloromethane followed by chromatography on alumina. **I** is determined by reduction, diazotisation and coupling with N-1-naphthylethylenediamine (*cf.* Averell and Norris, *Brit. Abstr. C*, 1949, 170); a modified procedure is used for opalescent soln. Diethyl *p*-nitrophenyl phosphate (**II**), *p*-nitrophenol, the emulsifier used in compositions of **I** and also various drugs that would interfere are retained on the alumina. Microgram amounts of **I** may be detected and distinguished from **II** by paper chromatography followed by reduction with $TiCl_3$ and reaction with Na naphthaquinonesulphonate or *p*-dimethylaminobenzaldehyde.

A. G. Cooper

3097. **Determination of T-1824 (Evans blue) [azovan blue] in lipaemia and haemolysis by a modified extraction method.** A. Tornberg (Univ. Med. Clin., Lund.). *Acta Med. Scand.*, 1958, **161**, 69–77.—Coloured plasma (1 ml) was shaken with 0·5 ml of 15% benzethonium chloride and after addition of acetone (3 ml) the mixture was centrifuged and the dye in the extract was determined photometrically at 620 mμ. Lipaemia or haemolysis did not affect the results. The plasma vol. of ten rabbits calculated by this method was 42·1 ± 4·4 ml per kg body-weight and the corresponding blood vol. was 62·3 ± 5·7 ml.

Nutr. Abstr. Rev.

3098. **Determination of glucose in fish blood. A modification of the Folin - Malmros micro-procedure.** L. R. Murrell and P. F. Nace (Dept. of Biol., McMaster Univ., Hamilton, Ont., Canada). *Canad. J. Biochem. Physiol.*, 1958, **36** (11), 1121–1124.—Methods in use for glucose determination in mammalian blood are unsatisfactory when applied to fish blood. This is due primarily to inadequate pptn. of proteins, and in the proposed method the ratio of blood to tungstic acid is 1:100 instead of the usual 1:10. Gum ghatti, the dispersive agent used in the original method (*cf.* Folin and Malmros, *J. Biol. Chem.*, 1929, **83**, 115), is replaced with advantage by sodium lauryl sulphate.

H. F. W. Kirkpatrick

3099. **Glucose test indicator.** Miles Laboratories, Inc. Brit. Pat. 808,742; date appl. 17.5.56. U.S.A., date appl. 9.6.55.—A composition for detecting glucose in urine and other body fluids comprises a mixture of glucose oxidase, peroxidase, a compound which undergoes a colour change in the presence of H_2O_2, *e.g.*, *o*-tolidine dihydrochloride or benzidine dihydrochloride, and a buffer (Na acetate) to maintain the pH of the mixture between 2 and 8. The amount of glucose present is indicated by the extent of the colour change. The composition can be applied to filter-paper strips to form indicator papers.

J. M. Jacobs

3100. **A method for the determination of phenylpyruvic acid in urine with some examples of its use in dietary treatment of phenylpyruvic oligophrenia.** A. Følling and S. Sydnes (Clin. Lab., Rikshospitalet, Oslo, Norway). *Scand. J. Clin. Lab. Invest.*, 1958, **10** (4), 355–358.—The qual. test for phenylpyruvic acid (**I**) with an iron salt has been modified for use as a quant. method. Urine is brought to a pH of ≃ 2·2 by the addition

of conc. HCl, and an aliquot (0·1 to 2·0 ml) is adjusted to 2·25 ml with 0·1 *M* glycine - HCl buffer (pH 2·2). The mixture is allowed to stand for 1 hr., after which 0·25 ml of 50% (w/v) aq. $(NH_4)_2SO_4.FeSO_4$ soln. is added. The extinction is then determined at 600 mμ, and re-determined after the reaction mixture has stood for a further 1 hr. By taking the difference of the two readings, the amount of **I** can be determined from a previously prepared standard curve. The second reading eliminates the effect of the variable amount of yellow colour produced by compounds other than **I**. The max. extinction is reached very quickly, and the colour fades rapidly, even at room temp.; urine samples are therefore stored at 0° before examination. As the vol. of urine approaches 2·0 ml, phosphates interfere, but the addition of MgO prevents this. The method is simple and suitable for routine use. D. B. Palmer

3101. **Determination of sialic acid in blood serum.** E. Turchetto and L. Cenciotti (Ospedale Maggiore, Bologna). *Boll. Soc. Ital. Biol. Sper.*, 1959, **35** (6), 248–251.—Five methods of determining sialic acid are briefly reviewed and that due to Hess *et al.* (*J. Clin. Invest.*, 1957, **36**, 449) is described in detail. In it, the sample (1 ml) of test serum is de-proteinised with 1 ml of 10% trichloroacetic acid and 0·4 ml of the supernatant liquid is heated for 30 min. in a boiling-water bath with 5 ml of a reagent consisting of glacial acetic acid (95 ml) and conc. H_2SO_4 (5 ml). The extinction of the cooled pink soln. is read at 530 mμ against a reagent blank. C. Rainbow

3102. **New colorimetric method for the determination of blood urea.** C. F. La Rosa. *Rev. Asoc. Bioquim. Argentina*, 1958, **23**, 228–231.—The sample is treated first with urease to convert urea into ammonia, and then with phenol and sodium hypobromite when an emerald green colour is formed, the extinction of which is measured with the use of a No. 62 filter and compared with standards. G. H. Foxley

3103. **Practical application of diacetyl monoxime to the determination of urea.** M. Girard and C. Dreux (Lab. Centr. de Biochem., Hôpital Lariboisière, Paris). *Ann. Pharm. Franç.*, 1958, **16** (9–10), 604–610.—The determination of urea by its colour reaction with diacetyl monoxime (**I**) in the presence of an oxidising agent is investigated with a view to the use of a suitable but non-toxic oxidant and to the stabilisation of the colour. Chloramine T (**II**) in 0·05% soln. is suitable as an oxidant and the colour attains its max. in 10 min. and remains stable for several hours in diffuse light without special precautions. The absorption max. is at 470 mμ, but the appearance of the calibration curve differs according to the photometer used. *Procedure*—Proteins are pptd. from the fluid by adding 1% aq. NaOH soln. and 5% aq. $ZnSO_4.7H_2O$ soln. After filtering or centrifuging, to 1 ml of the supernatant liquid is added 2 ml of H_2SO_4 (500 ml of conc. acid diluted to 1 litre) and 0·5 ml of 3% aq. **I** soln. and the tube is heated on the water bath for 10 min.; **II** (0·5 ml of 0·5% aq. soln.) is added and the extinction is measured after 10 min. at 470 to 475 mμ. Standard soln. of urea, and urine after dilution (1:200), are similarly treated. The results are comparable with those by the standard xanthhydrol method. E. J. H. Birch

3104. **Colorimetric method of determining unesterified fatty acids in plasma.** L. A. Carlson and L. B. Wadström (King Gustaf V Res. Inst., Stockholm, Sweden). *Scand. J. Clin. Lab. Invest.*, 1958, **10** (4), 407–413.—Heparinised plasma after separation at 4° is treated with $CHCl_3$ - methanol (2:1), boiled for a few minutes, cooled and filtered. The filtrate is shaken with 0·5 *M* acid sodium phosphate, and allowed to stand overnight for separation of the 2 phases. After concentration of the $CHCl_3$ layer, the mixture of phospholipids and unesterified fatty acids (**I**) is chromatographed on a column of activated silicic acid and Hyflo-Supercel (2:1), the phospholipids being eluted with methanol, and **I** with $CHCl_3$. To separate the mixture of **I**, the $CHCl_3$ eluate is evaporated to dryness, dissolved in ethanol - diethyl ether, and adsorbed on a column of Amberlite IRA-400 (OH form). The **I** are eluted with acid ethanol - diethyl ether. To determine **I**, the eluate is evaporated to dryness *in vacuo*, and the acid residue is dissolved in methanol. Treatment with thionyl chloride produces the methyl esters of **I**. After removal of the solvent the esters are dissolved in diethyl ether, treated with alkaline hydroxylamine soln., and the resulting mixture is evaporated to dryness *in vacuo*. The addition of 0·1 w/v ferric perchlorate in ethanolic $HClO_4$ produces a red colour, which is measured at 520 mμ. Blanks and standards are included in each series of determinations. The error is ± 2·0% for 0·5 to 4·0 micro-equiv. of **I**. Recoveries of 93 to 96% are obtained with acids with more than 14 C atoms. This method enables **I**, glycerides, phospholipids, and cholesterol to be determined on the same 2 ml of plasma, and it is highly specific for **I** with more than 14 C atoms. D. B. Palmer

3105. **New colorimetric method for determination of sphingosine base in liquids.** T. Sakagami (Dept. Biochem., Sapporo Univ. Med., Japan). *J. Biochem., Tokyo*, 1958, **45**, 313–317.—Sphingolipids in tissue were extracted by heating under reflux with ethanol and were hydrolysed with $Ba(OH)_2$, then with conc. HCl by the method of McKibbin and Taylor (*J. Biol. Chem.*, 1949, **178**, 17, 29). The hydrolysate was made alkaline and extracted with $CHCl_3$, and sphingosine base was dissolved in benzene and oxidised with lead tetra-acetate. Sphingosine was extracted with light petroleum and dissolved in glacial acetic acid and Schiff's reagent, then SO_2 soln. and *iso*amyl alcohol were added. The colour in the *iso*amyl alcohol was determined colorimetrically against an unoxidised blank. Values for sphingosine were—human serum 4·4, human brain 660, rat brain 640 and rat liver 11·3 mg per 100 g of fresh tissue. Nutr. Abstr. Rev.

3106. **Quantitative analysis of plasmalogens by paper chromatography.** G. V. Marinetti, J. Erbland and E. Stotz (Rochester Univ., N.Y., U.S.A.). *Biochim. Biophys. Acta*, 1959, **31** (1), 251–252.—After preliminary separation of lecithin and phosphatidylethanolamine fractions by chromatography on silicic acid and hydrolysis with acetic acid the resulting lysophosphatides are chromatographed on paper impregnated with silicic acid, and developed with di*iso*butyl ketone - acetic acid - water (40:25:3 or 40:20:3) at 0° to 5°. The chromatograms are dried and stained and appropriate spots are cut out and eluted separately with *N* HCl in methanol. The eluates are evaporated to

dryness and after wet oxidation of the residues P is determined by the molybdenum blue reaction.
W. H. C. SHAW

3107. **A method for multiple electrolyte analyses on small samples of nervous tissue.** A. Ames, III, and F. B. Nesbett (Dept. Biol. Chem. Surg., Harvard Med. School). *J. Neurochem.*, 1958, **3** (2), 116–126.—A system of analysis is described by means of which six of the major inorganic electrolytes can be determined on a single 17·5-mg sample of tissue. N. E.

3108. **Extraction of plant samples and the determination of total soluble carbohydrates.** R. P. Murphy (Dept. of Pathology, St. Finbarr's Hosp., Cork, Eire). *J. Sci. Food Agric.*, 1958, **9** (11), 714–717.—Total sol. carbohydrate in leaves and the uptake of sucrose by excised roots are rapidly determined with anthrone reagent. *Extraction*—Pack the freshly cut leaves (in polyethylene bags) in crushed solid CO_2 and store at − 18°. Macerate 10 g with 80% ethanol (40 ml) for 3 min., filter through No. 41 paper and wash the residue twice with 80% ethanol. Make up to 100 ml with ethanol; evaporate 1 ml of this soln. to dryness and take up in 25 ml of H_2O (in 5 aliquots) to separate the H_2O-insol. pigments. Make up to 30 ml with H_2O, filter, and take 1 ml for the test. *Procedure*—Chill 5 ml of reagent (1 g of anthrone in 475 ml of H_2SO_4 and 37·5 ml of H_2O) at 0° for 15 min., add quickly 1 ml of test soln., then shake for 7 sec. and chill again. Place in boiling H_2O for 10 min., then cool at 0° for 5 min. Measure the colour on an EEL colorimeter (filter 607), *vs.* a H_2O blank. Use glucose and sucrose to prepare standard graphs. Determine the uptake of sucrose by roots by immersing the sample in culture soln. at 26° in the presence of O and take hourly aliquots, measuring the diminishing concn. of sucrose by the anthrone reagent.
P. D. PARR-RICHARD

3109. **Quantitative determination of mannose, fucose and ribose on filter-paper.** J. W. Date (Dept. of Hygiene and Biochem., Univ., Aarhus, Denmark). *Scand. J. Clin. Lab. Invest.*, 1958, **10** (4), 444–445.—The method for the paper-chromatographic separation and determination of lactose, galactose, glucose, arabinose and xylose in biological fluids (*Anal. Abstr.*, 1959, **6**, 1014) is extended to include mannose, fucose and ribose. The chromatogram is sprayed with aniline - citric acid and heated, and the coloured spots are eluted with *n*-butanol - pyridine - water (2:2:1, by vol.). The extinction of the eluates is determined at 420 mμ; 15 to 800 μg of carbohydrate can be determined with an average recovery of 99%. The colour yields for these sugars relative to galactose are—mannose 0·67, fucose 0·55, and ribose 0·70.
D. B. PALMER

3110. **The enzymic synthesis of inositol phosphatide. [Determination of inositol.]** B. W. Agranoff, R. M. Bradley and R. O. Brady (Nat. Inst. of Neurol. Diseases and Blindness, Bethesda, Md., U.S.A.). *J. Biol. Chem.*, 1958, **233** (5), 1077–1083.—The method, which is based on that of Dixon and Lipkin (*Anal. Abstr.*, 1954, **I**, 2557), depends on oxidation with HIO_4 which is followed spectrophotometrically. Other sugars are first oxidised by heating with 0·3 *N* $Ba(OH)_2$, after which oxidation is effected, first in the cold with HIO_4 (for oxidation of glycerol) and then at 65° in the dark for 2 hr. Readings are taken at 260 mμ, and the difference between the two is a measure of the amount of inositol. J. N. ASHLEY

3111. **Determination of allantoin in comfrey root (*Symphytum officinale* L.).** F. Kaczmarek and A. Walicka. *Biul. Inst. Roślin Leczniczych*, 1958, **4** (4), 273–280.—The method is based on alkaline hydrolysis of the sample, followed by slightly acid hydrolysis, which yields glyoxylic acid. A red complex is formed from this acid and phenylhydrazine hydrochloride, the extinction of which is measured. *Procedure*—To 1 g of powdered root add 100 ml of water, and set aside the mixture for 2 hr. at room temp., stirring frequently. Filter through paper and transfer a 1-ml aliquot of the clear filtrate to a 25-ml flask. Add 1 ml of 0·5 *N* NaOH and immerse the flask in boiling water for exactly 7 min. to hydrolyse allantoin to allantoic acid, then cool the flask to 20° and add 2·5 ml of 0·5 *N* HCl and 1 ml of phenylhydrazine hydrochloride soln. (33%). Immerse the flask in boiling water for 2 min., then cool immediately in salt and ice for 15 to 30 min. until the contents begin to freeze. Add 3 ml of conc. HCl (previously cooled) and 1 ml of $K_3Fe(CN)_6$ and shake for 30 min. The glyoxylic acid - phenylhydrazine complex is oxidised to give a red colour. Make up the soln. to 25 ml with water and measure the extinction against a water blank, using an S53 filter and 1-cm cell. W. ROUBO

3112. **Chromatographic identification of anthocyanin pigments.** J. B. Harborne (John Innes Hort. Inst., Hertford, England). *J. Chromatography*, 1958, **1** (6), 473–487.—The chromatography of anthocyanins is reviewed and some previously unpublished R_F values are given and discussed. The value and limitations of the chromatographic approach to anthocyanin identification, and some of the methods used for studying the occurrence and distribution of anthocyanins in plants are described. (67 references.) G. P. COOK

3113. **Ooporphyrin in the shells of hen and other eggs and its quantitative determination.** L. Schwarz, W. Dicket and H. Ketel (Hygien. Inst., Hamburg). *Hoppe-Seyl. Z.*, 1958, **312** (1–3), 37–44.—Procedures are described for isolating the porphyrins of powdered egg-shell as their methyl esters and for their quant. determination. The yield is ≃ 11 mg per kg of dark-brown shells, but it varies much from egg to egg and is related to the colour.
F. POWELL

3114. **O-Phosphoserine phosphatase from baker's yeast. [Determination of O-phosphoserine.]** M. Schramm (Div. of Nutr. and Physiol., Publ. Health Res. Inst., New York, U.S.A.). *J. Biol. Chem.*, 1958, **233** (5), 1169–1171.—A specific phosphoserine phosphatase, isolated from baker's yeast, is used to hydrolyse L-O-phosphoserine to serine and inorganic phosphate, and the phosphate is determined by any of the standard methods. L-Serine, adenosine triphosphate, and DL-phosphothreonine do not interfere. J. N. ASHLEY

3115. **Determination of histamine in blood and urine by adsorption on Amberlite IRC-50 with special reference to the normal values for histamine in human blood, and an analysis of the specificity of the method.** H. Dunér and B. Pernow (King Gustaf V Res. Inst., Stockholm, Sweden). *Scand. J. Clin. Lab. Invest.*, 1958, **10** (3), 233–239.—The

authors' method (*Ibid.*, 1956, **8**, 296) is used for urine, and in a slightly modified form for serum. A resin column (10 mm × 80 mm) is used to adsorb the histamine, which is then eluted initially with *N* HCl followed by 0·01 *N* HCl. After neutralisation, the eluate is freed from salts by treatment with ethanol - acetone at − 15°, and assayed by means of isolated guinea-pig ileum. Dihydroergotamine is added to the organ bath to remove the effects of any 5-hydroxytyramine in the eluates. Administration of the antihistamine promethazine complicates the bio-assay of urinary eluates, but this compound can be removed by extraction with diethyl ether before adsorption on the resin column. It is also shown that oral promethazine has no effect on the urinary excretion of histamine. The average recovery of histamine is 94%, and results are reported for serum (0·6 to 2·8 μg, mean 1·29 ± 0·17, per 100 ml of blood) and for blood cells (1·0 to 4·6 μg, mean 2·66 ± 0·33, per 100 ml of blood). D. B. PALMER

3116. **Extraction of ninhydrin-developed spots from paper chromatograms and determination of amino acids.** G. S. Robbins and W. J. Olson (Barley and Malt Lab., Agric. Res. Service, U.S. Dept. of Agric.). *Proc. Amer. Soc. Brew. Chem.*, 1957, 60–65.—In the procedure developed, the choice and regulation of operating details to obtain max. colour intensity and, therefore, sensitivity of detection, were investigated. A simple apparatus (illustrated) was devised in which an amino-acid spot was supported above refluxing ethanol, the colour being continuously extracted with fresh solvent. Inclusion of $Cu(NO_3)_2$ in the ethanol produced the same type of stable salmon-pink compound for each of the 25 amino acids and derivatives tested. This permitted spectrophotometric measurement to be made at the wavelength of max. absorption, 510 mμ, common to almost all the separately extracted substances. Sensitivity values are tabulated for 25 amino acids and derivatives. K. R. C.

3117. **Paper chromatography of the copper complexes of amino acids.** M. Beck, J. Császár and T. Huszka (Inst. Inorg. Anal. Chem., Univ., Szeged, Hungary). *Magyar Kém. Foly.*, 1958, **64** (11), 438–439.—The interaction of amino acids during their chromatography can be eliminated by the use of their complexes with Cu^{2+}. The ascending technique is applied, with phenol or *o*-cresol saturated with water at 27°. The metal component is detected with a 0·2% ethanolic soln. of dithiooxamide or 8-hydroxyquinoline, which also liberates the amino acids, which are then detected with ninhydrin. The R_F values for 13 complexes are given. G. SZABO

3118. **Some factors in ultra-violet densitometry of amino-acid chromatograms.** H. ven Horst, H. Tang and V. Jurkovich (Dept. of Chem., Marycrest College, Davenport, Iowa). *Anal. Chem.*, 1959, **31** (1), 135–138.—The factors considered were—the effect of irrigating solvents on the fluorescence of the filter-paper, the grades of paper most suitable for uniform background and max. amino-acid fluorescence, the effect of time and temp., the sensitivity of the amino-acid fluorescence in u.v. compared with the ninhydrin reaction in the visible spectrum, the stability of the fluorescence of the developed chromatogram, the amino acids which fluoresce, and the reproducibility of results. The fluorescence of solvents such as phenol, cresol and pyridine renders them unsuitable, and certain grades of paper are unsatisfactory because of irregular backgrounds. Heating the chromatograms for 10 min. at 120° produces max. fluorescence of the amino acids and this is stable under atmospheric conditions. Sensitivity and reproducibility are comparable with visible-range methods; 27 amino acids have been observed to fluoresce. D. W. Moss

3119. **A new method for the determination of lysine in the presence of other basic amino acids.** J. C. Sanahuja and D. S. Rios (Cátedra de Bromatología, Fac. de Farm. y Bioquím., Buenos Aires). *Rev. Asoc. Bioquim. Argentina*, 1958, **23**, 232–237.—Histidine passes through Amberlite IRC-50 resin (pH 7·0), whilst arginine and lysine are adsorbed. These two compounds are eluted from the column with 0·1 *N* HCl and passed down an Amberlite IRA-400 column which adsorbs the lysine only. Elution with an acetate buffer (pH 4·5) washes the lysine from the column, diazotised *p*-nitroaniline is added and the extinction is measured at 520 mμ. The mean recovery on samples containing 400 to 1100 μg of lysine was 99%. G. H. FOXLEY

3120. **Micro-determination of hydroxyproline with chloramine T and *p*-dimethylaminobenzaldehyde.** H. Stegemann (Med. Forschungsanst., Max-Planck Gesellschaft, Göttingen). *Hoppe-Seyl. Z.*, 1958, **311**, 41–45.—The method depends on the oxidation of hydroxyproline by chloramine T at pH 6 to yield pyrrole, which is determined in terms of the red colour it gives with *p*-dimethylaminobenzaldehyde in the presence of $HClO_4$. Other amino acids do not interfere. NUTR. ABSTR. REV.

3121. **Determination of glutamic acid, glutamine, glutathione and γ-aminobutyric acid and their distribution in brain tissue.** S. Berl and H. Waelsch (N.Y. State Psychiatric Inst.). *J. Neurochem.*, 1958, **3** (2), 161–169.—The four compounds are separated on columns, converted into γ-lactones by treatment with nitrous acid and then to their corresponding hydroxamic acids (Sachs and Brand, *J. Amer. Chem. Soc.*, 1954, **76**, 3601). Glutathione forms a stable complex with *p*-chloromercuribenzenesulphonic acid. This, together with glutamic acid, is adsorbed on an alumina column. Glutamine and γ-aminobutyric acid present in the eluate are separated and purified by adsorption and elution from Dowex 50. Glutamic acid is eluted from the alumina column with 0·5 *N* acetic acid and subsequently the glutathione is eluted with 0·5 *N* aq. NH_3. N. E.

3122. **Rapid quantitative horizontal paper chromatography at an elevated temperature: determination of aspartic and glutamic acids.** H. R. Roberts and M. G. Kolor (Nat. Dairy Prod. Corp., Oakdale, Long Island, N.Y., U.S.A.). *Nature*, 1959, **183**, 460–461.—The method previously presented (*Anal. Abstr.*, 1958, **5**, 1626) is adapted to direct photometric determination on paper chromatograms developed for 2 hr. at 60° in phenol saturated with pH 12 buffer. H. F. W. KIRKPATRICK

3123. **Chromatography of arginine-containing peptides on a carboxylic acid resin.** T. Ando, S. Ishii and M. Kimura (Tokyo Univ., Japan). *Biochim. Biophys. Acta*, 1959, **31** (1), 255–256.—Separations are carried out on Amberlite IRC-50 (XE-64) ion-exchange resin eluted at 30° with

0·1 *M* sodium borate buffer (pH 8·0), in which the concentration of Na^+ is increased stepwise by the addition of 0·1 to 1·5 *N* NaCl. Recovery of total peptides is 99%. W. H. C. SHAW

3124. **Quantitative paper-chromatographic analysis of aromatic amino acids (tyrosine and phenylalanine) in the hydrolysate of a small quantity of a radioactive protein.** R. Munier (Inst. Pasteur, Paris). *J. Chromatography*, 1958, **1** (6), 524–533 (in French).—When small amounts of radioactive proteins are hydrolysed with HCl, a part of the tyrosine is converted into 3-chlorotyrosine. Constant values for the tyrosine content of a protein can be obtained only if this is taken into account. G. P. COOK

3125. **Titration of bovine serum albumin in urea and in formaldehyde solutions.** M. Levy (Dept. Biochem., College of Dentistry, Univ., New York). *Compt. Rend. Lab. Carlsberg, Sér. Chim.*, 1958, **30** (16), 301–308.—The titration of bovine serum albumin (BSA) in urea does not increase the number of proton-accepting groups at the acid end-point, but increases the apparent proton affinity of the carboxylate groups. Results of formol titrations give more accurate measures of the amino and iminazole groups of BSA than are hitherto available. N. E.

3126. **Determination of serum albumin based on the binding of bromophenol blue.** S. H. Blondheim (Rothschild-Hadassah Med. Sch., Jerusalem, Israel). *Bull. Res. Council Israel, E*, 1957, **6**, 223–224.—To 10 ml of dye-buffer reagent (100 ml of a citrate buffer plus 1 ml of a 0·1% aq. bromophenol blue soln., kept in a dark bottle in a refrigerator and prepared fresh every week), 0·02 ml of serum is added with a haemoglobin pipette rinsed in the serum to be determined, and mixed by inversion. The soln. is read within a few minutes in a Klett colorimeter with filter 59. Standards are prepared by adding 0·04, 0·06, 0·08 and 0·10 ml of a 1% albumin standard soln. to tubes containing 10 ml of the dye-buffer reagent, corresponding to serum albumin concn. of from 2 to 5 g per 100 ml. Readings are plotted and used as a reference curve. CHEM. ABSTR.

3127. **Electrophoresis of serum proteins in mixed agar gels.** S. A. Karjala (M. D. Anderson Hospital and Tumour Inst., Texas). *Makromol. Chem.*, 1958, **28** (2), 103–113 (in English).—The literature on electrophoresis in gels is reviewed. In the experiments carried out with two-phase gels, the cathode side of the plate was covered with 1·5% agar soln. (5 ml) and the anode side with starch-agar gel (5 ml) containing 2% of starch; the electrode strips were each coated with the respective gels (1 ml) and the samples placed in holes at the interface between the two gels. A greater spread of protein spots was obtained than with either of the single-phase runs. In studies on electro-endosmotic mobility, the neutral substance selected was glyceraldehyde with barbitone and phosphate buffers. Both substances were spotted by layering the plates dropwise with an ammoniacal $AgNO_3$ soln. This was allowed to stand for 1 to 2 min. on the plates, which were then washed, treated with 6 *N* aq. NH_3 soln., and washed again. Immuno-electrophoretic studies were made on large-scale runs by teasing strips of the gels on to glass plates which were then treated with 1·5% agar in physiological saline soln. A long channel was cut in the agar, parallel to the strips of gel, and filled with a mixture of equal parts of rabbit anti-normal-human serum and warm agar soln. The plates were placed in a closed, humid chamber, until arcs of antigen-antibody ppt. had formed, and then leached for 3 days in saline soln., immersed in 70% ethanol for 1 hr. to dry, and stained with azocarmine. The increase in separation of the various arcs was noted by comparison of the run in agar with that in the two-phase 2% starch-agar and agar plate. The electro-endosmotic effect in the electrophoresis of proteins in agar gel was inhibited by the use of agar gels containing gelatinised starch, but could not be eliminated completely. The most suitable starch concn. was 2%. K. R. C.

3128. **Calcium phosphate chromatography of normal human serum and of electrophoretically isolated serum proteins.** S. Hjertén (Uppsala Univ., Sweden). *Biochim. Biophys. Acta*, 1959, **31** (1), 216–235.—A study is reported of the separation of serum proteins on calcium phosphate (hydroxyapatite) columns eluted with sodium or potassium phosphate buffers (pH 6·8), the concn. of which are increased stepwise from 0·02 to 1·3 *M*. The concn. of proteins in the fractions so obtained from whole serum and from serum proteins isolated by zone electrophoresis is measured by the extinction at 280 mμ, and the fractions are then further examined by free or paper electrophoresis or by ultra-centrifugation. The separation is achieved of β-lipoprotein and γ-globulin from whole serum and of caeruloplasmin from α_2-globulin. W. H. C. SHAW

3129. **Quantitative determination of proteinuria by Closs's polythionate method.** T. Lygren and H. E. Refsum (Centr. Lab., Oslo City Hosp., Norway). *Scand. J. Clin. Lab. Invest.*, 1958, **10** (4), 415–417.—The reliability of the Closs method (*Ibid.*, 1957, **9**, 349) was investigated, and the results were shown to compare favourably with those of the micro-Kjeldahl method, whilst both methods gave higher results than the Esbach method. Ketone bodies and increased bile excretion do not affect the method, but increased polypeptide excretion does. It is concluded that this simple method is both reliable and suitable for routine use. D. B. PALMER

3130. **Chromatographic separations of cytosine-containing compounds.** P. Reichard (Dept. of Chem., Karolinska Inst., Stockholm, Sweden). *Acta Chem. Scand.*, 1958, **12** (10), 2048.—A column of Dowex 50 (H^+ form, 200 to 400 mesh) (10 cm × 0·9 cm) is used, fractions of 4 to 5 ml per 30 min. being collected. The chromatogram is developed first with 0·2 *N* acetic acid and then with *N* HCl. Paper chromatography may also be used with a borate-containing solvent [5 *M* ammonium acetate, pH 9·5 (20 ml), saturated $Na_2B_4O_7$ soln. (80 ml), ethanol (220 ml) and 0·5 *M* EDTA (0·5 ml)]. This solvent may also be used for separation of uracil- or thymine-containing compounds. H. F. W. KIRKPATRICK

3131. **Elimination of substances interfering with nucleic acid estimation.** M. De Deken-Grenson and R. H. De Deken (Bruxelles Univ., Belgium). *Biochim. Biophys. Acta*, 1959, **31** (1), 195–207.—Substances that interfere with the determination of nucleic acids, by u.v. absorption and by the orcinol colorimetric reaction for pentoses, may be removed by adsorption of the ribonucleotides on Dowex-2 ion-exchange resin. Adsorption is carried

out from 0·025 *M* tri(hydroxymethyl)methylamine - HCl buffer (pH 7·8) by contact with 1 mg of resin for each 1 to 2 μg of ribonucleotides, and is followed by elution with *N* HCl. Results so obtained are compared with those given by established purification techniques and by other colorimetric methods.
W. H. C. SHAW

3132. **Determination of Δ^5-3β-hydroxysteroids.** G. W. Oertel and K. B. Eik-Nes (Dept. of Biochem., Coll. of Med., Univ. of Utah, Salt Lake City, U.S.A.). *Anal. Chem.*, 1959, **31** (1), 98–100.—When 0·5 to 20 μg of any Δ^5-3β-hydroxysteroid is dissolved in 2·5 ml of a reagent composed of 1 vol. of 95% ethanol (freshly distilled *in vacuo* over Ag_2O) and 2 vol. of conc. H_2SO_4 a yellow colour is developed with max. absorption at 405 mμ (400 mμ for dehydro*epi*androsterone). The colour reaches its max. intensity after 2 min., then begins to fade after 2 hr. The max. absorbance at 405 mμ can be corrected by applying the formula—corrected absorbance at 405 mμ is equal to the observed absorbance at 405 mμ less one-half the sum of the absorbancies at 380 and 430 mμ. For 10 μg of various steroids dissolved in 2·5 ml of the reagent, the absorbance is of the order of 0·110 to 0·258. The sensitivity of the reaction depends on the ethanol to acid ratio, max. absorbance being obtained with the 1:2 composition. The presence of steroids with configurations other than Δ^5-3β-hydroxy does not seem to affect the development of the colour, and when assayed under similar conditions several of these steroids have been shown to give no absorbance max. between 380 and 430 mμ. The reaction can be used for the determination of Δ^5-3β-hydroxysteroids in biological material.
D. W. Moss

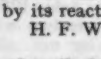
3133. **Chromatographic separation of oxytocin and vasopressin on carboxymethylcellulose.** A. V. Schally, H. S. Lipscomb and R. Guillemin (Baylor Univ. Med. Coll., Houston, Tex., U.S.A.). *Biochim. Biophys. Acta*, 1959, **31** (1), 252–254.—The separation is carried out on carboxymethylcellulose equilibrated with 0·01 *M* ammonium acetate (pH 4·5), and is followed by elution with 0·02 *M* ammonium acetate (pH 6), graded to 0·2 *M* (pH 7). The significance of the elution patterns obtained is discussed.
W. H. C. SHAW

3134. **Simplified method for the routine measurement of urinary oestriol.** W. B. Eberlein, A. M. Bongiovanni and C. M. Francis (Children's Hosp., Philadelphia, Pa., U.S.A.). *J. Clin. Endocrin. & Metabolism*, 1958, **18** (11), 1274–1284.—Urine is extracted with diethyl ether after enzyme hydrolysis of steroid conjugates. The dried extract is dissolved in *N* NaOH and the soln. is autoclaved at 10 lb pressure for 5 min., cooled in running water and washed by shaking with diethyl ether, which is discarded. The aq. soln. is acidified with *N* HCl and extracted with diethyl ether, the ether soln. is washed with alkali and water and the ether is removed. The extract is chromatographed on alumina with 2% ethanol in benzene, the column is washed with 4% ethanol in benzene and the oestriol is eluted with 30% ethanol in benzene. After removal of the solvent the steroid is determined fluorimetrically by its reaction with H_2SO_4.
H. F. W. KIRKPATRICK

3135. **A comparison of methods for the analysis of oestrone, oestradiol and oestriol in extracts of human urine.** T. F. Gallagher, S. Kraychy, J. Fishman, J. B. Brown and G. F. Marrian (Sloan-Kettering Inst. for Cancer Res., New York). *J. Biol. Chem.*, 1958, **233** (5), 1093–1096.—Satisfactory agreement is obtained between all the analytical results when oestrone, oestradiol and oestriol are determined by the following methods after administration of [16-^{14}C]17β-oestradiol—(*i*) reverse isotopic dilution, (*ii*) determination of the radioactivity of the peak tubes after counter-current distribution, (*iii*) the methods of Brown (*Anal. Abstr.*, 1955, **2**, 3161) and Brown *et al.* (*J. Endocrin.*, 1957, **16**, 49) in which the Kober colour reaction is used, and (*iv*) determination of the radioactivity of the separated oestrogen methyl ether fractions obtained by the method of Beer and Gallagher (*J. Biol. Chem.*, 1955, **214**, 351).
J. N. ASHLEY

3136. **New method for the chemical determination of oestrogenic hormones in urine.** G. Ittrich (Hormonlab. d. Univ.-Frauenklinik der Charité, Berlin). *Hoppe-Seyl. Z.*, 1958, **312** (1–3), 1–14.—The Kober reaction for phenols, which is satisfactory for the determination of pure oestrogenic hormones, is not so with urine extracts except when these are comparatively rich in oestrogens. By using H_2SO_4 diluted with 2% quinol to a suitable concn., which varies with the particular substance to be determined, and extracting the coloured complex with 2% *p*-nitrophenol in $CHCl_3$, the sensitivity and specificity are much improved. Quantities of 0·2 to 20 μg may be determined colorimetrically or 0·005 to 0·5 μg fluorimetrically. Recoveries were 95 to 105% for oestrone, 92 to 103% for oestradiol and 70 to 90% for oestriol.
F. POWELL

3137. **Quantitative determination of progesterone and pregnanediol in human plasma.** I. F. Sommerville and G. N. Deshpande (Chelsea Hosp. for Women, London). *J. Clin. Endocrin. & Metabolism*, 1958, **18** (11), 1223–1236.—Plasma is shaken with 0·33 *N* NaOH and extracted with dichloromethane - diethyl ether (1:4). The solvent is removed and the dry extract is dissolved in *n*-hexane and chromatographed on alumina, the column being washed with *n*-hexane and then with 5% $CHCl_3$ in hexane. Progesterone (**I**) is eluted with 20% $CHCl_3$ in hexane, and pregnanediol (**II**) with $CHCl_3$ alone. **I** is determined by its reaction with isoniazid, and **II** by the H_2SO_4 reaction. Conjugated **II** is determined similarly after enzyme hydrolysis.
H. F. W. KIRKPATRICK

3138. **Infra-red spectra of ring-D steroid lactones. II. Structure - absorption correlations.** H. Rosenkrantz and C. Gual (Worcester Found. for Exptl. Biol., Shrewsbury, Mass., U.S.A.). *Spectrochim. Acta*, 1959, **13** (4), 291–295.—The spectra of a number of ring-D steroid lactones have been examined and found to have three absorption frequencies characteristic of such molecules. These bands are near 1219 cm^{-1}, 1110 cm^{-1} and 989 cm^{-1}, and were correlated with a D-homolactone structure. Other bands enable individual lactones to be identified. Acetate derivatives show the 1110-cm^{-1} band strongly, but the 989-cm^{-1} band is weak, while the 1219-cm^{-1} band is masked by the acetoxy absorption. The 1219-cm^{-1} band arises from the  grouping, and the 1110-cm^{-1} band from the group.
H. M.

3139. **Steroid conjugates. II. Separation and identification of further 17-oxosteroid glucuronides from urine by means of combined electrophoretic and chromatographic methods.** H. Pelzer, W. Staib and D. Ott (Physiol.-Chem. Inst. d. Med. Akad., Düsseldorf). *Hoppe-Seyl. Z.*, 1958, **312** (1–3), 15–21.—Details are given for chromatography on alumina columns, paper chromatography and high-voltage paper electrophoresis of 17-oxosteroid conjugates, the steroids being located by the Zimmermann reaction or u.v. absorption. The value of successive application of the three processes is stressed, and examples are given of the results obtained with each. F. Powell

3140. **Determination of conjugated 17-hydroxycorticosteroids in human peripheral blood.** F.-E. Krusius and M. Oka (Centr. Lab., Kivelä Hosp., Helsinki, Finland). *Scand. J. Clin. Lab. Invest.*, 1958, **10** (4), 359–363.—To 5 ml of heparinised plasma are added 0·5 ml of 1·0 *M* KH_2PO_4, pH 4·5, 400 units of bacterial β-glucuronidase suspended in 1 ml of re-distilled water, and 0·3 ml of aqueous 5% (w/v) $NaHSO_3$ soln. This mixture (pH 6·0 to 6·2) is then incubated in a stoppered tube for 48 hr. at 37° in the dark. The liberated plasma 17-hydroxycorticosteroids (**I**) are extracted with 25 ml of pure re-distilled dichloromethane and determined by means of the Silber - Porter reaction according to the method of Peterson *et al.* (*cf. Anal. Abstr.*, 1957, **4**, 2341). Simultaneously, the free **I** are determined on another 5 ml of plasma. The difference between the two results gives the amount of conjugated **I**. Standards (1 μg of cortisol per 5 ml) and blanks (water) are included in each series of determinations. The $NaHSO_3$ is shown to be most effective in removing chromogens which interfere with the Silber - Porter determination, and it also eliminates any effects due to solvent impurities. This method is simple, and avoids laborious purification processes. D. B. Palmer

3141. **Spectrophotometric determination of traces of phenolic steroids in 3-oxosteroids.** M. Legrand, V. Delaroff and R. Smolik (Res. Lab., Roussel-Uclaf, Paris). *J. Pharm. Pharmacol.*, 1958, **10** (11), 683–686.—Steroids of the oestradiol type cannot be determined in the presence of a large excess of 3-oxosteroids by direct u.v. spectroscopy, because of masking of the phenolic band at $\simeq 280$ mμ. The interference is almost completely removed by reduction of the ketone with potassium borohydride (**I**); the residual absorption of the reduction products is slight and nearly linear in the range 272 to 300 mμ, and is corrected by a graphical method. *Procedure*—Dissolve the sample (500 mg containing $< 0{\cdot}02\%$ of phenol or 200 mg containing $< 1\%$ of phenol) in methanol (35 ml) and add a soln. of **I** (400 mg or 160 mg) in 0·1 *N* NaOH (4 ml). After 6·5 hr. at room temp., add *N* HCl (4 ml), shake the mixture and dilute to 50 ml with methanol. Record the spectrum of the steroid soln. against a similarly prepared blank in cells of path length 10 cm (or 5 cm if the phenol content is $> 0{\cdot}5\%$). Oestradiol (1 mg) in testosterone (200 mg) was determined with a coeff. of variation of $\pm$ 2%, estimated from 14 measurements. A. R. Rogers

3142. **Differentiation of Δ^4-3-oxosteroids and $\Delta^{1,4}$-3-oxosteroids with *iso*nicotinic acid hydrazide [isoniazid].** L. L. Smith and T. Foell (Lederle Lab. Div., American Cyanamid Co., Pearl River, N.Y.). *Anal. Chem.*, 1959, **31** (1), 102–105.—Two forms of the reagent are used—(*i*) a dil. ethanolic soln. (0·5 g) of isoniazid is dissolved in 1 litre of re-distilled ethanol and 0·625 ml of conc. HCl is added) (**I**), and (*ii*) a conc. methanolic soln. (4 g of isoniazid is dissolved in 1 litre of re-distilled methanol and 5 ml of conc. HCl is added) (**II**). Three procedures are available for the visualisation of $\Delta^{1,4}$-3-oxosteroids on paper chromatograms; in each case Δ^4-3-oxo compounds are detected initially by spraying the chromatograms with **I** and are visible as yellow fluorescent spots in u.v. light or in daylight after drying. *Procedure A*—The paper is sprayed with methanol - conc. HCl (40: 1) and the $\Delta^{1,4}$-3-oxosteroids become visible after 16 to 24 hr. as yellow fluorescent spots in u.v. light and as yellow spots in daylight. *Procedure B*—The papers are dipped into **I** for 2 min., dried at room temp. for 16 to 24 hr. and the $\Delta^{1,4}$-3-oxo compounds appear as yellow spots as before. *Procedure C*—Spraying with **II** reveals the $\Delta^{1,4}$-3-ketones after 3 to 16 hr. at room temp. When the untreated chromatogram is dipped into **II**, both classes of compound appear as yellow spots in daylight and as yellow fluorescent spots in u.v. light. When the untreated papers are sprayed directly with **II** the Δ^4-3-oxosteroid spots appear in 2 min. and those of $\Delta^{1,4}$-3-oxosteroids in 3 to 16 hr. The sensitivity of detection of unsaturated 3-ketones by procedure *C* extends to submicrogram levels. The reagents are stable up to 1 week. The subsequent use of tetrazolium reagents for the detection of reducing α-oxosteroids is not affected. D. W. Moss

3143. **Infra-red absorption spectra of α-halogenated oxosteroids.** F. Meda (Res. Lab. of S.A. Farmitalia, Milan, Italy). *Spectrochim. Acta*, 1958, **13** (1–2), 75–79.—A further study of 3-oxo-4-halogeno-Δ^4-steroids is made (*cf.* Camerino *et al.*, *Farmaco, Ed. Sci.*, 1956, **11**, 586). The frequencies of the i.r. absorption bands of α-halogenated ketosteroids in the carbonyl and double-bond region, and the frequency shift on halogenation are tabulated. The introduction of an α-halogen atom raises the conjugated 3-ketone band (normally at 1661 cm^{-1}) to 1889, 1683 and 1681 cm^{-1} for derivatives containing F, Cl and Br, respectively. The influence of the halogen substituent on C=C frequencies is studied; the greatest frequency shift is produced by Br and the least by F. Absorption curves of halogenated progesterone, testosterone, cortisone and deoxycortone are shown. The results permit the characterisation and differentiation of epimer steroids according to the frequency shifts induced by substituents in equatorial or axial positions. B. B. Bauminger

3144. **Detection of saponins and sapogenins on paper chromatograms by Liebermann - Burchard reagent.** G. R. Van Atta and J. Guggolz (Western Reg. Res. Lab., U.S. Dept. of Agric., Albany, Calif.). *J. Agric. Food Chem.*, 1958, **6** (11), 849–850.—For the location of legume saponins and sapogenins on paper chromatograms, the papers are drawn through Liebermann - Burchard reagent (equal vol. of H_2SO_4 and acetic anhydride) at 60° to 70°, and spread on stainless-steel surfaces. Characteristic colours reach optimum intensity in about 15 min., by which time very little background colour has developed. The chromatograms can be accurately recorded on coloured photographic slides. The colours obtained are—red with abietic, cholic, echinocystic and ursolic acids, erythrodiol, and soyasapogenol B; purple with cholesterol, sitosterol, and smilagenin; magenta with oleanolic acid, soyasapogenol C, and uvaol;

and blue with castanogenin, hederagenin and medicagenic acid. Amounts from 5 to 10 μg were detected. M. D. ANDERSON

3145. **Simple procedure for the routine assay of *Dioscorea* tubers.** M. P. Morris, B. A. Roark and B. Cancel (Fed. Expt. Sta., Agric. Res. Service, U.S. Dept. of Agric., Mayaguez, Porto Rico). *J. Agric. Food Chem.*, 1958, **6** (11), 856–858.—A method is developed for routine determinations of diosgenin (**I**), based on maceration of the fresh tuber with water, refluxing with 2 *N* HCl to liberate **I** from the saponin dioscin and to dissolve most of the starchy tuber, extraction of the **I** from the residue by light petroleum, and partial removal of solvent; **I** crystallises out, and is washed, dried, and weighed. One operator can assay 12 to 24 samples a day, and results are within ± 5% of those by a more accurate method.
M. D. ANDERSON

3146. **A new method for peroxidase determination.** W. M. Hunting, M. Gagnon and W. B. Esselen (Dept. Food Technol., Univ. of Massachusetts, Amherst). *Anal. Chem.*, 1959, **31** (1), 143–144.—The time for the appearance of colour after the addition of H_2O_2 to a spot of the peroxidase soln. and guaiacol (or guaiacum resin soln.) on a paper disc is measured. The method is calibrated against known peroxidase concn., the log. of time (sec.) having a linear relationship with the log. of enzyme concn. Some practice is required in the detection of the first trace of colour, and highly coloured substances may interfere. Good reproducibility is obtained over the concn. range of 1×10^{-3} to 1×10^{-6}% by wt. of peroxidase with guaiacol and over the range 1×10^{-4} to 1×10^{-6} % by wt. with guaiacum resin. D. W. Moss

3147. **New method for catalase determination.** M. Gagnon, W. M. Hunting and W. B. Esselen (Dept. Food Technol., Univ. of Massachusetts, Amherst). *Anal. Chem.*, 1959, **31** (1), 144–146.—Paper discs saturated with various dilutions of enzyme soln. are allowed to fall into H_2O_2 soln. in wide test-tubes. The time elapsing between the entry of the disc into the soln. and the instant at which it again reaches the surface (due to the release of O) is measured. A linear relation holds between the log. of time (sec.) and the log. of the enzyme concn. A blank determines the time of rise of the disc without catalase activity. Reproducibility is good over the range of concn. 10^{-3} to 10^{-4} % by wt. of pure enzyme; conditions must be carefully standardised in quant. work.
D. W. Moss

3148. **Simplification of Thunberg's method for determining the activity of dehydrogenases.** E. Macovschi and N. Moldoveanu (Inst. Biochem., Acad. R.P.R., Bucharest). *Rev. Chim., Acad. R.P.R.*, 1958, **3** (2), 191–193 (in English).—A branched test-tube with a rubber stopper is used. The mouth of the tube is plugged with a roll of filter-paper soaked in alkaline pyrogallol. After 30 min. the oxygen-free tube is inclined so that the enzyme and buffer soln. in one branch of the tube mix with the methylene blue and substrate soln. in the other branch and the decoloration time is measured from the moment of mixing. The results are in good agreement with those obtained with the usual Thunberg tubes.
K. R. C.

3149. **Studies in histochemistry. II. Microdetermination of hyaluronidase and its inhibition by fractions of isolated mast cells.** D. Glick, R. Ottoson and P. R. Edmondson (Histochem. Lab., Dept. of Physiol. Chem., Univ. of Minnesota, Minneapolis, U.S.A.). *J. Biol. Chem.*, 1958, **233** (5), 1241–1243.—The method of Tolksdorf *et al.* (*Brit. Abstr. A*III, 1949, 1290) for the turbidimetric determination of hyaluronidase is adapted for a micro-method which requires only one-hundredth to one-thousandth of the usual amount of sample. J. N. ASHLEY

3150. **Colorimetric estimation of some cholinergic esters. Application to the demonstration of acetylcholinesterase activity.** J. S. Hanker, A. Gelberg and B. Witten (Chem. Res. Div., U.S. Army Chem. Warfare Lab., Army Chem. Center, Md.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1958, **47** (10), 728–730.—Acetylcholine (**I**), methacholine and succinylcholine, but not carbachol or bethanechol, give a positive Schönemann reaction which may be used for the quant. analysis of the first 3 of these esters. Anticholinesterase activity and its inhibition may be demonstrated by use of the reaction for the determination of residual **I**. *Procedure*—To the sample soln. (1 ml) add a 0·1% soln. of *o*-dianisidine in acetone (3 ml), 3% aq. H_2O_2 (1 ml) and 0·05 *M* aq. Na_3PO_4 (1 ml), and after 4 min. measure in a Beckman Model C colorimeter with filter No. 44. A. R. ROGERS

See also Abstracts—**2825**, Clinical chemistry (review). **2869**, Separation of ^{137}Cs in biological material. **2924**, Determination of P. **3020**, Separation of carbohydrates by gas - liquid chromatography. **3021**, Titration of glucose. **3023**, Titration of carbohydrates. **3024**, Detection of sorbitol. **3029**, Determination of alginic acid. **3046**, Determination of $-OCH_3$ groups in phenolic compounds in man. **3164**, Determination of oestrogens. **3195**, Determination of lipase in milk. **3199**, Determination of amino acids and amino acid deoxyfructose in apricots. **3200**, Determination of chlorophyll and pheophytin in broccoli. **3308–3312**, Electrophoresis of proteins.

Pharmaceutical analysis

3151. **Fluorescence of powdered vegetable drugs under ultra-violet radiation.** C. J. Kokoski, R. J. Kokoski and F. J. Slama (Dept. of Pharmacognosy, Univ. of Maryland, U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1958, **47** (10), 715–717.—A table is given of the characteristic fluorescence of 131 powdered vegetable drugs and 2 powdered animal drugs in u.v. light after treatment with the following reagents—*N* methanolic NaOH, *N* aq. NaOH, *N* aq. HCl, HNO_3 - H_2O (1:1), H_2SO_4 - H_2O (1:1).
A. R. ROGERS

3152. **Determination of morphine in poppy capsules. III.** W. Poethke and E. Pechmann (Friedrich Schiller Univ., Jena, Germany). *Pharm. Zentralh.*, 1958, **97** (10), 463–467.—The method of Poethke and Arnold (*Pharm. Zentralh.*, 1949, **88**, 1; 1951, **90**, 145) has been improved. The morphine in the material obtained by extraction with dichloromethane is dissolved in methanol and tartaric acid in aq. soln., purified by passage through a column of Al_2O_3 and determined with chlorodinitrobenzene.
A. R. ROGERS

3153. **Determination of narcotine and papaverine by infra-red spectrophotometry and non-aqueous titration.** L. Kum-Tatt, R. A. Rockerbie and L. Levi (Gov. Chem. Dept., Singapore). *J. Pharm. Pharmacol.*, 1958, **10** (10), 621–624.—The carbonyl group of the lactone ring present in narcotine (**I**) shows strong i.r. absorption at 1760 cm^{-1} in $CHCl_3$ or CCl_4 soln. The intensity of the band, corrected for background absorption, is directly proportional to the concn. of **I** up to 0·25%. The amount of papaverine (**II**) plus **I** may be determined by titration with $HClO_4$ in glacial acetic acid (*cf.* Levi *et al.*, *Bull. Narcotics*, 1953, **5**, 15), and the content of **II** in a mixture is obtained by difference.
A. R. ROGERS

3154. **Paper-chromatographic separation of hyoscyamine and norhyoscyamine.** R. E. A. Drey (Wellcome Chem. Works, Dartford, Kent, England). *J. Pharm. Pharmacol.*, 1959, **11** (1), 64.—To separate oscine, hyoscine, meteloidine, hyoscyamine, norhyoscyamine, tigloidine and apoatropine, develop the alkaloid salts with H_2O-saturated *n*-butanol or *iso*butyl alcohol by descending chromatography on Whatman No. 1 paper impregnated with 0·025 *M* oxalic acid. Valeroidine runs with norhyoscyamine.
A. R. ROGERS

3155. **Application of electrophoresis to some difficult cases of separation of alkaloids (purine bases, atropine - brucine - strychnine, cocaine - morphine).** R. Paris and G. Faugeras (Lab. de Mat. Med., Fac. Pharm., Paris, France). *Ann. Pharm. Franç.*, 1958, **16** (5), 305–310.—Three series of bases that are difficult to separate by paper chromatography are investigated with a view to separation by electrophoresis. The effect of pH is investigated from pH 2·5 to 12 with solutions of comparable ionic strength adjusted with formic acid and aq. NH_3. The electrophoresis is carried out with a p.d. of 800 V on 20 cm of Arches 301 paper (40 V per cm) and a current density of 1 mA per cm (over 6 cm width). The concn. of the original soln. is 0·2% in ethanol for the alkaloids and 0·5% in 10% aq. NH_3 for the purine bases, and samples of 2·5 µl are used. The alkaloids are displaced towards the cathode in acid soln. and the purine bases are displaced with good separation towards the anode in alkaline soln. Morphine alone behaves as a true amphoteric compound and has an isoelectric point at pH $\simeq$ 10. The effect of concn. of the electrolyte is investigated and though the speed of migration increases with dilution, the sharpness of the spots decreases. The effect of alteration of the applied voltage is also investigated; at too low a voltage the speed of migration is balanced by the counter-current of soln. due to evaporation loss, so that 800 V across the 20 cm of paper is necessary. The most difficult to separate are brucine and strychnine, but a separation of 10 mm between the spots is attained in 15 min. at 800 V. The recommended conditions are—for purine bases, pH $>$ 11, 800 V for 1 hr.; for morphine - cocaine, pH $\simeq$ 6 or $>$ 11 (under the alkaline conditions there is loss of sharpness of the cocaine spot); for atropine - strychnine - brucine, pH 3 and a low ionic strength (1% formic acid).
E. J. H. BIRCH

3156. **Determination of the alkaloid content of ergot.** J. Bayer (Anal. Lab., Pharm. Works, Kőbánya, Budapest). *Magyar Kém. Foly.*, 1958, **64** (11), 429–432.—A routine method, suitable for assaying the ergotoxine, ergotamine and water-soluble alkaloid contents of ergot, is described. The alkaloids are liberated with MgO and extracted with diethyl ether. The ether soln. are extracted with 1% tartaric acid. The water-insol. alkaloids are then removed with benzene at pH 5·3. The benzene soln. are transferred to an Al_2O_3 column, which adsorbs the alkaloids. Ergotoxine is eluted first with 1% ethanolic $CHCl_3$ and then ergotamine with 4% ethanolic $CHCl_3$. The two fractions are extracted with tartaric acid soln. and the extinctions are measured at 317 mµ, giving the ergotoxine and ergotamine contents. The extinction of the original tartaric acid extract before and after treatment with benzene gives the total and water-soluble alkaloid contents. The separation of the alkaloids is tested by model experiments.
G. SZABO

3157. **Micro-crystal tests for colchicine.** C. C. Fulton (Div. Microbiol., Food and Drug Admin., Dept of Health, Educ. and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (4), 756–758.—When 5% chloroauric acid in H_2SO_4 (1 + 1) or H_3PO_4 (2 + 1) is added to colchicine (**I**) on a microscope slide, brown rosettes of birefringent needles are formed. Branching needles may be obtained from **I** with a reagent prepared from $HAuCl_4.4H_2O$ (1 g), 40% HBr (1·5 ml) and H_3PO_4 (3 + 1 or 1 + 1) to 25 ml. Iodine in KI soln. added to a soln. of **I** (neutralised when necessary with $NaHCO_3$) produces slightly birefringent, almost colourless needles and plates.
A. A. ELDRIDGE

3158. **Paper chromatography of the alkaloids of *Rauwolfia serpentina*.** A. Hameed Khan (Central Lab., Pakistan C.S.I.R., Karachi). *Pakistan J. Sci. Ind. Res.*, 1958, **1** (3), 194–196.—The ascending chromatographic technique of Axelrod and Bandurski (*J. Biol. Chem.*, 1951, **193**, 405) was used. A suitable amount of alkaloid in $CHCl_3$ was applied to one corner of a Whatman paper No. 1. The paper was formed into a cylinder, stapled together with fine threads and suspended by a hook inside a bell-jar. By lowering the hook, the paper dipped into a solvent system. Chromatograms were allowed to run until the solvent almost reached the upper edge of the paper (about 5 hr.). The air-dried chromatograms were viewed under u.v. light. The addition of methanol to the solvent appeared to affect the movement of all the bases in such a way that a wider range of R_F values was obtained and the spots became rounder and more compact, resulting in a better and cleaner separation of the alkaloidal mixtures.
K. R. C.

3159. **Extraction and fluorescence of some oxidation products of reserpine.** E. B. Dechene (Charles E. Frosst & Co., Montreal, Canada). *J. Amer. Pharm. Ass., Sci. Ed.*, 1958, **47** (10), 757–758.—In the fluorimetric method of assay of reserpine (**I**) in pharmaceutical preparations, oxidation products of **I** are either eliminated in the preliminary extraction and washings or, if present, cause little interference. By the method described, **I** can be assayed in the presence of its oxidation products as accurately as by the nitrite absorptiometric method. The need for a blank correction in the nitrite method is confirmed.
A. R. ROGERS

3160. **Qualitative and quantitative chromatographic technique for the determination of the more important cardiac glycosides in the leaves of *Digitalis purpurea* L.** B. Borkowski and J.

Lutomski. *Biul. Inst. Roślin Leczniczych*, 1958, **4** (4), 299–309.—Samples of strospeside, gitoxin, digitoxin and gitaloxin have been used as standards for a qual. chromatographic separation of extracts from *D. purpurea* leaves, with S. & S. paper 2043b-mgl impregnated with a mixture of formamide and methanol (1:3). The sample is macerated with water for 24 hr., and the aq. extract is extracted with $CHCl_3$. An aliquot (≡ 0·15 to 0·2 g of sample) is developed by the ascending technique with *p*-xylene - ethyl methyl ketone (1:1) for 4 hr., the chromatogram is dried for 15 min. at 80° to 100°, then treated with a mixture (15:1) of 25% ethanolic trichloroacetic acid and 3% aq. chloramine T. The coloured spots are identified under u.v. light. For a quant. determination, standard and sample soln. are developed, the chromatogram is cut into zones containing the separated and identified glycosides, and these are washed with Baljet reagent (5 ml of 10% NaOH soln. plus 95 ml of 1% aq. picric acid) to elute strospeside and with xanthhydrol reagent (10 mg in 100 ml of acetic acid plus 1 ml of 30% HCl) to elute digitoxin and gitaloxin. The eluates are measured photometrically, with suitable filters and cells. W. Roubo

3161. **Determination of lanatosides A, B, C and D in the leaves of *Digitalis lanata* Ehrh. after chromatographic separation.** B. Borkowski and Z. Kowalewski. *Biul. Inst. Róslin Leczniczych*, 1958, **4** (4), 311–314.—Extract 5 g of well-ground leaves twice for 1 hr. with 50 ml of 80% methanol, finally washing the leaves with 20 ml of 80% methanol. To the combined extracts and washings add 10 ml of 10% Pb acetate soln., make up to 125 ml with methanol, and filter through paper. To 100 ml of the filtrate (≡ 4 g of sample) add 10 ml of water and extract with CCl_4 (1 × 50 ml, 2 × 25 ml). Add 50 ml of water to the methanol layer and extract with $CHCl_3$ (3 × 50 ml). Dry the $CHCl_3$ extract with anhyd. Na_2SO_4, and filter. Evaporate the $CHCl_3$ under vacuum at 35° and dissolve the residue in methanol - $CHCl_3$ (1:1) (1 ml). Chromatograph aliquots (0·01 to 0·03 ml) of standard and test soln. on S. & S. 2043b or Whatman No. 1 paper that has been impregnated with formamide - methanol (1:3) and dried at room temp. for 15 min. For the mobile phase, mix 70 ml of $CHCl_3$, 20 ml of dioxan and 5 ml of *n*-butanol, add formamide slowly until a slight opalescence appears, then set the mixture aside for 15 min., and use the lower layer. Develop the chromatogram by the descending technique for 2 to 3 hr. and dry it at 105° for 15 min. Treat the zones containing the standard soln. with a freshly prepared mixture (15:1) of 25% ethanolic trichloroacetic acid soln. and 3% aq. chloramine-T soln., then dry the chromatogram at 120° for 15 min. and identify and mark the coloured spots under a u.v. lamp. Cut out the corresponding zones containing the test soln. separated into lanatosides A, B, C and D, place each in a test-tube and add xanthhydrol reagent (10 ml of xanthhydrol dissolved in 100 ml of ice-cold water, plus 1 ml of 36% HCl). Shake, and immerse the tubes in boiling water for 5 min., then cool to room temp., and determine the extinctions with a Pulfrich photometer (0·5-cm cells and an S53 filter). Refer the results to calibration curves. W. Roubo

3162. **Determination of khellin in the fruits of *Ammi visnaga* (L.) Lamarck.** B. Borkowski and W. Raszejowa. *Biul. Inst. Róslin Leczniczych*, 1958, **4** (4), 330–338.—The method described is based on the modified Fahmy procedure (*cf.* Supniewska, *Dissert. Pharm.*, 1956, **8**, 63). Weigh 0·25 g of a powdered sample (0·6 mm mesh) and boil with 50 ml of water for 30 min. Filter through paper and boil again with 25 ml of water for 15 min. Filter and wash the residue with 5 ml of boiling water. After cooling, extract the combined filtrates and washings with $CHCl_3$ (4 × 25 ml). Wash the $CHCl_3$ extract with 10 ml of water, dry the $CHCl_3$ extract with 5 g of anhyd. Na_2SO_4 and evaporate it to dryness. Dissolve the dry residue in 3 ml of H_3PO_4 (d = 1·30) and warm on a water bath for 5 min. Filter through paper and dilute 1 ml of clear filtrate with 3 ml of H_3PO_4. Measure the extinction with a Pulfrich photometer (S47 filter and a 0·5-cm cell) and determine the khellin content from a standard curve. W. Roubo

3163. **Colorimetric determination of anthracene derivatives: application to rhubarb grown in France.** R. Paris and H. Moyse. *Ann. Pharm. Franç.*, 1958, **16** (9–10), 561–574.—An examination was made of methods for determining anthracene derivatives in purgative drugs, based on the measurement of the red colour produced in alkaline soln. by free anthraquinones; anthracene derivatives in the form of glycosides are first hydrolysed, and those in reduced form (anthranols and anthrones) are oxidised. The material must be dried and finely powdered. The effects of variations in operating conditions are discussed. The methods do not give absolute values, but satisfactory comparative results can be obtained under standard conditions. Rhubarb (*Rheum palmatum* var. *tanguticum*) cultivated in France contained about the same amount of total anthracene derivatives as that from Holland, and nearly as much as samples from China. M. D. Anderson

3164. **Estimation of oestrogen and its derivatives in pharmaceutical preparations.** A. C. Roy (Central Drug Res. Inst., Lucknow, India). *Indian J. Pharm.*, 1958, **20** (11), 321–324.—Prepare a soln. of the oestrogen (1 mg) in 95% ethanol (5 ml), add 1 ml of Millon's reagent (5 g of mercuric nitrate and 20 ml of fuming HNO_3 with H_2O to 100 ml), heat for 10 min. at 50°, cool and measure against a reagent blank at 420 mμ. The yellow colour is stable for some hours. To determine oestradiol in tablets, prepare the soln. by extraction of the powdered sample with 95% ethanol in a Soxhlet apparatus. For oestradiol dibenzoate, saponify the sample, acidify and extract with benzene; evaporate an aliquot of the extract to dryness and dissolve the residue in alcohol. For oestradiol *cyclo*pentylpropionate in sesame oil soln., dissolve the sample in light petroleum, extract with methanolic KOH, acidify and extract with benzene; evaporate an aliquot to dryness and dissolve the residue in alcohol. A. R. Rogers

3165. **Determination of novobiocin in the presence of *iso*novobiocin.** A. A. Forist, S. Theal and W. A. Struck (Dept. of Phys. and Anal. Chem., The Upjohn Co., Kalamazoo, Mich.). *Anal. Chem.*, 1959, **31** (1), 100–102.—The 3-O-carbamoylnoviose (**I**) formed by acid hydrolysis of novobiocin is determined by periodate oxidation; the 2-O-carbamoylnoviose formed simultaneously from *iso*novobiocin does not interfere. A 40 to 50-mg sample of the material is treated with 5 ml of anhyd. trifluoroacetic acid in a glass-stoppered flask and is set aside in the dark for 2 hr. Water

(20 ml) is slowly added and the ppt. is separated by centrifugation and filtration through sintered glass. Standard novobiocin samples (20, 40 and 60 mg) are carried through the same procedure. A 20-ml aliquot of each filtrate is oxidised with 5 ml of 0·2 *N* HIO_4 (standardised weekly with 0·1 *N* sodium arsenite and 0·01 *N* iodine) in the dark for 1 hr. (A reagent blank is similarly treated.) Partial neutralisation with 2·5 ml of 10 *N* NaOH is completed by slow addition of $NaHCO_3$ until a saturated soln. is produced. Sodium arsenite (0·1 *N*) (10 ml) and 2 ml of KI soln. (5%) are added, and after 15 min. the excess of arsenite is titrated to a starch end-point with 0·01 *N* iodine. A 99·9% recovery has been obtained on 20 determinations with pure novobiocin (standard deviation 1·5%), and on 14 novobiocin - *iso*novobiocin mixtures results were accurate to ± 1·5%.

D. W. Moss

3166. **Microbiological assay of nystatin by turbidimetry.** J. Philippe, J. Teillon and M. Craipeau (S.I.F.A. Res. Lab., Paris). *Ann. Pharm. Franç.*, 1958, **16** (9–10), 578–585.—The fungicidal compound nystatin, produced by *Streptomyces noursei*, is extracted from fermentation liquors, pharmaceutical preparations, etc., by 70% *iso*propyl alcohol, and determined by adding 0·2 ml of each of 4 dilutions of the extract to tubes containing 9·8 ml of nutrient solution inoculated with *Saccharomyces cerevisiae*; these are incubated with shaking at 37° for 4 hr., the cells are then killed with formaldehyde, and the light transmission is measured at 550 mμ, and expressed as a percentage of that of an uninoculated tube. The results are compared with a standard curve. The relation between log. concn. of nystatin and transmission (%) is linear in the range 3 to 7·5 units per ml. The precision of the method is ± 4·75%. The results agreed well with those obtained by a method using the diffusion of nystatin from filter-paper discs on an agar medium inoculated with *S. cerevisiae*.

M. D. Anderson

3167. **Identification of substituted barbituric acids by X-ray diffraction.** P. P. Williams (Dominion Lab., Wellington, New Zealand). *Anal. Chem.*, 1959, **31** (1), 140–143.—The X-ray diffraction patterns are presented of 20 commonly used 5:5-disubstituted barbituric acids, crystallised from ether and analysed on a Geiger counter diffractometer.

K. A. Proctor

3168. **Determination of barbiturates with a cation exchanger.** E. B. L. M. van Nispen tot Pannerden (St. Elisabeths Gasthuis, Arnhem, Netherlands). *Pharm. Weekbl.*, 1958, **93** (22), 987–989.—The sample (200 to 250 mg), dissolved in 70% ethanol (10 ml), is added to a column containing Amberlite IR-120 (H) resin covered with 70% ethanol, and eluted with 70% ethanol (4 × 10 ml) at the rate of one drop per sec. The eluate is mixed with 25 ml of demineralised H_2O and titrated with 0·1 *N* NaOH, with thymolphthalein as indicator. Allobarbitone and aprobarbital are determined bromimetrically after evaporating the ethanol from the eluate. If the sample contains Na_2CO_3, the CO_3 must be removed by gentle heating after ion exchange. The results are in good agreement with those obtained by the official methods of the Dutch (6th Ed.) and British (1958) Pharmacopoeias.

G. Burger

3169. **Microchemical differentiation between optical isomers of N-methylmorphinan analgesics.** E. G. C. Clarke (Royal Vet. Coll., London, England). *J. Pharm. Pharmacol.*, 1958, **10** (10), 642–644.—When 5% Na_2CO_3 soln. (0·1 μl) is added to a 0·2 to 1% soln. of (±)-3-hydroxy-N-methylmorphinan in 1% HCl (0·1 μl), crystals in the form of bunches of small plates appear in 30 min.; under similar conditions, the (+)- or (−)-isomers give only amorphous ppt. To distinguish between the (+)- and (−)-forms, mix the sample (0·1 μl) with a 1% soln. of a known isomer (0·1 μl) and 5% Na_2CO_3 soln. (0·1 μl) and examine for crystals. A similar method is used to distinguish between the optical isomers of 3-methoxy-N-methylmorphinan; the racemic mixture gives rosettes of crystals with saturated soln. of trinitrobenzoic acid, but the (+)- or (−)-forms give only oily amorphous ppt.

A. R. Rogers

3170. **An analytical property of amyleine [amylocaine] and sparteine.** F. Gélébart (Lab. Dausse, Usine de Morigny-Champigny, Seine-et-Oise, France). *Ann. Pharm. Franç.*, 1958, **16** (9–10), 574–578.—Amylocaine (**I**) and sparteine (**II**) can be distilled in steam from alkaline soln. Attempts to determine **I** either by determination of the alkali consumed or by distilling into standard acid lead to errors owing to hydrolysis of the benzoic ester group, but it may be characterised by the following test. **I** hydrochloride (0·1 g) is dissolved in 50 ml of water and made alkaline with 0·5 ml of 5% NaOH soln., and 25 ml is distilled into a mixture of 1 ml of HCl and 5 ml of H_2O. The usual reactions for alkaloids give a positive test with the contents of the receiver, but not with those of the distillation flask after acidification. **II** is not so easily attacked by NaOH and the procedure can be applied quant. *Procedure*—A sample of **II** (0·3 to 0·4 g) is dissolved in 75 ml of water and 25 ml of 0·1 *N* NaOH is added; 75 ml is distilled over in 25 min. While the contents of the flask are still warm, 25 ml of 0·1 *N* H_2SO_4 is added, and after boiling for 2 or 3 min., the excess of acid is titrated with 0·1 *N* NaOH, to phenolphthalein. The base can be recovered unchanged from the distillate. In this method the base behaves as if it were dibasic, while in the direct titration it is monobasic and gives a poor end-point. A modification of the gravimetric tungstosilicate method is also proposed.

E. J. H. Birch

3171. **Analysis of phenylephrine and tetracaine [amethocaine] by filter-paper chromatography.** H. Schriftman (Wyeth Laboratories, Radnor, Pa.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1959, **48** (2), 111–113.—Develop 5 to 25-μg quantities of amethocaine hydrochloride (**I**) and phenylephrine hydrochloride (**II**) on Whatman No. 1 paper with the upper phase of *n*-butanol - acetic acid - H_2O (5:1:3) by the descending technique. After 20 hr., detect **I** at R_F 0·83 by a modified Dragendorff reagent spray, and detect **II** at R_F 0·63 by a spray reagent containing diazotised sulphanilic acid. Analyse the colour spots by quant. photo-electric densitometry. Breakdown products of **II** with R_F values of 0·58 (main product), 0·26 and 0·37, respectively, cause no interference.

A. R. Rogers

3172. **Assay of promazine and its separation from chlorpromazine and promethazine.** L. Cavatorta (Anal. Dept., Lepetit S. p. A., Milan, Italy). *J. Pharm. Pharmacol.*, 1959, **11** (1), 49–53.—To determine promazine (**I**), chlorpromazine

(**II**) or promethazine (**III**), add acetate buffer of pH 2·0 (5 ml) and a 0·1% soln. of $PdCl_2$ in aq. HCl (0·5 ml) to the aq. sample (1 ml containing $\simeq$ 100 μg of **I**, **II** or **III**); dilute to 7 ml with H_2O, stir, and after 15 min. measure the extinction at 500 mμ against a reagent blank. To determine **II** or **III** in the presence of **I**, add buffer soln. (5 ml) and $PdCl_2$ soln. (0·5 ml) to the aq. sample (2 ml containing $\simeq$ 500 μg of **II** or **III**) and extract with ethyl acetate (10 ml); wash the organic phase with *N* HCl (5 ml) and after 5 min. measure the extinction of the organic phase at 440 mμ. To determine **III** in the presence of **I** and **II**, add mercuric sulphate reagent [HgO (5 g) dissolved in conc. H_2SO_4 (80 ml) and H_2O (20 ml)] (1 ml) to the aq. sample (1 ml containing 50 to 100 μg of **III**), heat on a bath of boiling water for 10 min., cool, and dilute to 5 ml with H_2O; measure the extinction at 500 mμ after 10 min. A. R. ROGERS

3173. **The analysis of phenothiazine derivatives with tranquillising properties.** J. Milne (Dept. of Nat. Health and Welfare, Ottawa, Ontario, Canada). *J. Amer. Pharm. Ass., Sci. Ed.*, 1959, **48** (2), 117–122.—Non-aqueous titration methods have been compared with the tungstosilicic acid gravimetric method of Blazek and Stejskal (*Pharmazie*, 1956, **12**, 27) and with the control procedures used by the pharmaceutical manufacturers, which are described. Pecazine can be titrated in hexane - acetone (2:1) or in acetone, acepromazine and fluopromazine in acetone, and perphenazine, prochlorperazine and thiopropazate in benzene - nitromethane (2:1), with $HClO_4$ in dioxan as titrant in all instances. The gravimetric and titration methods have been modified to permit the assay of pharmaceutical preparations of these compounds. A. R. ROGERS

3174. **Identification of meprobamate by adsorption chromatography on chromatoplates.** A. Fiori and M. Marigo (Ist. Med. Legale e Assicurazioni, Univ., Padova). *Nature*, 1958, **182**, 943–944.—Transfer the sample of meprobamate to the starting line of a chromatoplate of silicic acid - rice starch (*cf.* Demole, *Anal. Abstr.*, 1958, **5**, 3197; Reitsema, *Anal. Abstr.*, 1954, **1**, 2469) and develop for 2 hr. with *cyclo*hexane - ethanol (17:3) as ascending solvent. Dry the plate at room temp., spray lightly with conc. H_2SO_4, heat at 110° to 115° for 2 or 3 min., spray lightly with H_2O and reheat until yellow spots due to meprobamate appear at R_F $\simeq$ 0·3. For quant. determination, suspend the cut-out spot in H_2O (1 ml), add 0·2% quinol in conc. H_2SO_4 (1 ml), heat on a boiling-water bath for 20 min. and measure at 420 mμ. A. R. ROGERS

3175. **Quantitative determination of methylal in formalin.** O. Heggelund and K. Ruzicka (Gullaug Kjemiske Fabrikker A/S, Lillestrøm, Norway). *Anal. Chem.*, 1959, **31** (1), 138–139.—Mix the sample (100 ml) with powdered Na_2SO_3 (50 g), add methanol (100 ml), distil slowly through a fractionating column and collect the distillate in a 50-ml flask cooled to − 15°. Weigh the distillate, titrate a 1-g aliquot with Karl Fischer reagent and adjust the water content of the bulk of the distillate to exactly 1%. Mix, measure the refractive index at 18° and calculate the concn. of methylal in the adjusted distillate from a calibration curve. A. R. ROGERS

3176. **Estimation of 4-aminosalicylic acid and isoniazid in mixtures.** P. C. Bose, T. Sen and G. K. Ray (Central Drug Res. Inst., Lucknow, India). *Indian J. Pharm.*, 1958, **20** (11), 324–327.—The 4-aminosalicylic acid (**I**) is separated from the isoniazid (**II**) and determined by titration with alkali; the residual **II** is determined by the method of Kidani *et al.* (*Bull. Nat. Hyg. Lab., Tokyo*, 1954, **72**, 95). *Procedure*—For mixtures containing **II** (50 mg) and the Na or Ca salt of **I** (2 g), dissolve the sample in H_2O (25 ml), add conc. HCl (5 or 10 ml), warm for 15 min. and filter; wash the ppt. with dil. HCl (6 × 10 ml). Dissolve the ppt. in ether, wash the soln. with H_2O and combine the washings with the previous filtrate and washings. Evaporate the ether soln. to dryness, dissolve the residue in acetone (100 ml) and titrate a 10-ml aliquot with 0·1 *N* NaOH, with bromothymol blue as indicator. Neutralise the combined aq. layers with $NaHCO_3$, dilute with H_2O to 500 ml and mix a 1-ml aliquot with 2% alcoholic *p*-dimethylaminobenzaldehyde soln. (2 ml) and *iso*butyl alcohol (12 ml); after 10 min., add 1% oxalic acid soln. (1 ml), dilute to 17 ml with *iso*butyl alcohol and measure the extinction at 409 mμ after a further 20 min. For mixtures containing **I** and **II**, extract the sample directly with ether, evaporate, and titrate the residue of **I**; determine **II** in an aq. extract of the ether-insol. residue. A. R. ROGERS

3177. **Enzymatic spectrophotometric determination of sucrose in U.S.P. syrups.** J. P. Comer and H. F. Brickley (Anal. Dept., Eli Lilly and Co., Indianapolis). *Anal. Chem.*, 1959, **31** (1), 109–110.—The syrup is diluted so as to contain $\simeq$ 4 mg of glucose per ml after hydrolysis by heating with HCl (6 vol. of 20%). After cooling the hydrolysate and adjusting the pH to 5·6, an aliquot, with blanks and glucose standards, is treated with a glucose oxidase - horse-radish peroxidase - anisidine reagent for exactly 10 min. The reaction is stopped by adding HCl (1:1), and the extinction of the soln. is determined at 400 mμ. The glucose content, and hence the sucrose, is calculated by reference to the standards. If a mixture of glucose and sucrose is being assayed, the colour formation before hydrolysis is also measured. The standard soln. must be set aside for at least 1 hr. to equilibrate the α- and β-forms, the enzyme being specific for β-D-glucose. The average difference between the actual concn. and that found by determination for 10 syrups was − 0·2%, and the coeff. of variation of 5 replicates was ± 0·93%. D. W. Moss

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See also Abstracts—**2825**, Pharmaceuticals and related drugs (review). **2922**, Determination of isoniazid. **3045**, Isotope dilution analysis of salicylic acid and acetylsalicylic acid. **3067**, Specifications for essential oils. **3071**, Determination of cineole in eucalyptus oil. **3093**, Determination of 4-aminosalicylic acid in blood, etc. **3095**, Determination of nitrofurantoin in urine. **3111**, Determination of allantoin in comfrey root. **3133**, Separation of oxytocin and vasopressin. **3191**, **3192**, Determination of antibiotic residues in fish. **3196**, Detection of antibiotics in milk.

Food

3178. **Infra-red methods for the rapid determination of the moisture content of foods.** F. Schierbaum. *Ernährungsforschung*, 1958, **3** (4), 554–566.—A review is presented covering general principles, the

merits of various types of i.r. lamps, and published data in which the results obtained by i.r. drying of different foods are compared with those obtained by standard oven-drying methods; operating details are given, including wt. of sample, distance from the source of irradiation, and the power of the lamps used. The saving of time effected by i.r. drying amounts to 65 to 90% of the corresponding time for oven-drying. When (as with glucose monohydrate) the decomposition phase overlaps or closely follows the dehydration phase, conditions may often be improved by increasing the distance between the sample and the i.r. lamp.

P. S. ARUP

3179. **The determination of sugars in foods.** A. Rouquette (Hôpital Mil. de Val-de-Grâce, Paris, France). *Ann. Falsif.*, 1958, **51**, 325–329.—The methods of calculation of Ruiz (*cf. Anal. Abstr.*, 1954, **2**, 1030, **1335**) are adapted for use with the Bertrand method of sugar determination. For a mixture of two reducing sugars, if V is the total vol. (ml) of 0·1 N $KMnO_4$ used in the titration, p_1 and p_2 are the respective weights (mg) of the two sugars, and A and B are the respective weights (mg) of the two sugars corresponding to V according to the Bertrand sugar tables, then $p_1/A + p_2/B = 1$, whence, knowing $p_1 + p_2$, the weights of the individual sugars can be calculated. Mixtures of glucose and sucrose can be calculated according to the usual relation $S = (R - r) \times 0{\cdot}95$, where S is the weight (mg) of sucrose and r and R are the reducing powers before and after inversion; when the reducing powers of glucose and invert sugar are approx. equal, *i.e.*, when the total sugar in the sample is 40 to 60 mg, $G/A + D/B = 1$, where G is the glucose, determined by reduction before inversion, D is the invert sugar, and A and B are the values for glucose and invert sugar, respectively, corresponding to the vol. (ml) of $KMnO_4$ soln. required after inversion. For mixtures of more than two sugars, an equation holds of the type $p_1/A + p_2/B + p_3/C + \ldots = 1$. The precision of the result depends on the precision of the reducing technique employed. Results are quoted for mixtures of glucose and galactose and of glucose and sucrose.

R. E. ESSERY

3180. **Studies on the relationship between thiocyanate formation and the goitrogenic properties of foodstuffs. I. The preformed thiocyanate content of some foods.** N. Michajlovskij and P. Langer (Endocrin. Inst. d. Slowak. Akad. der Wissenschaft., Pressurg, Czechoslovakia). *Hoppe-Seyl. Z.*, 1958, **312** (1–3), 26–30.—The thiocyanate content of several foodstuffs was investigated by two techniques, one depending on the colour given with Fe, the other by conversion into cyanogen bromide and treatment with benzidine in pyridine. The methods usually agreed within 10%, but sometimes the difference was much greater. Oxalate, salicylate, keto acids, phenols, tannin, etc., all give a colour with Fe, while tyrosine, tryptophan and cyanide give the second reaction. The discrepancies are attributed to varying concn. of these or similar interfering substances. In some cases it was impossible to determine the thiocyanate content by either technique owing to the colour of the original foodstuff. The highest values were found in the brassica vegetables whose goitrogenic properties are well recognised.

F. POWELL

3181. **Photometric determination of the amylose content of starch by the "blue value" method.** S. Augustat. *Ernährungsforschung*, 1958, **3** (4), 567–574.—After consideration of published data, the following procedure has been selected. Mix the sample (100 mg, dry basis) with N NaOH (6·66 ml) and allow to stand, with occasional stirring, for 24 hr. Dilute to $\simeq$ 1·5 litres with vigorous shaking, adjust to pH 5, and add an aq. soln. containing 0·5% of iodine and 0·75% of KI (20 ml). Dilute to 2 litres, and dilute 25 ml of this soln. to 150 ml. Measure the extinction of the final soln. at 644 to 700 mμ (OG2 filter) and at $\simeq$ 20° in a Lange colorimeter with a 34·5-mm cell. Prepare a calibration curve for extinctions for the range from 0% of amylose plus 100% of amylopectin to 100% of amylose plus 0% of amylopectin. A constant voltage must be maintained for the colorimeter lamp. The max. error is $\Rightarrow$ 2% of amylose.

P. S. ARUP

3182. **Determination of copper and iron in starch and starch syrups.** G. Tegge and W. Kempf (Bundesforschungsanst. f. Getreideverarbeit., Detmold, Germany). *Stärke*, 1958, **10** (10), 243–247.—The main difficulty in the method of Lindemann (*cf. Anal. Abstr.*, 1956, **3**, 3191) is the complete destruction of organic matter before photometric analysis of metals. Dry ashing is found to result in some loss of Cu and Fe. Complete removal of organic substances is achieved by heating the sample (2 g) in a mixture of conc. HNO_3 (20 ml), conc. H_2SO_4 (5 ml) and 70% $HClO_4$ (8 ml). The temperature is raised until all acids have been removed by boiling (15 min.). Copper and Fe are then determined spectrophotometrically with Na diethyldithiocarbamate and 1:10-phenanthroline, respectively. It is shown that this procedure does not cause any loss of Cu or Fe.

E. DUX

3183. **Quantitative determination of reducing sugar with 2:3:5-triphenyltetrazolium chloride.** K. Amako. *Proc. Res. Soc. Japan Sugar Refineries' Technologists*, 1958, **7**, 65–69.—The method of Carruthers and Wootton (*cf. Anal. Abstr.*, 1956, **3**, 234) is slightly modified to allow for the effect of reducing substances produced by the degradation of sucrose in hot alkaline soln. The modifications comprise heating the tube containing the test soln. for 6 min. at 80° instead of 90 sec. at 100°, and diluting the sample with ethanol to 5 times its original vol. The red colour produced is measured spectrophotometrically at 490 mμ. The results obtained showed good reproducibility and good agreement with those obtained by Ofner's method.

SUGAR IND. ABSTR.

3184. **Automatic water determination in cereal products using the Karl Fischer reagent.** W. Seibel and H. Bolling (Bundesforschungsanst. f. Getreideverarbeit., Detmold, Germany). *Getreide u. Mehl*, 1958, **8** (10), 73–76.—A fully automatic electrometric titration method is described. The burette delivering the Karl Fischer reagent is magnetically controlled and the end-point is detected by the "dead stop" technique, and is indicated by a continuously-shining red light. Determinations were made on 0·5-g aliquots of finely ground samples of a variety of cereals, flours, doughs and bread, and gave results in good agreement with those of oven-drying methods.

J. V. RUSSO

3185. **Improvements in and relating to the measurement of moisture content of granular materials.** National Research Development Corp.

[Inventor: T. A. Oxley]. Brit. Pat. 807,993; date appl. 23.10.54.—The moisture content of cereal grains contained in sacks or bins, bales of hay, straw, etc., is determined by means of a device comprising a series of pointed, rod-like electrodes, for insertion in the material, arranged into two groups insulated from one another and connected to a hand-operated current generator and a resistance meter. J. M. JACOBS

3186. **Rapid method for the determination of fat in cereals.** D. B. West and A. F. Lautenbach (J. E. Siebel Sons' Co., Inc., Chicago). *Proc. Amer. Soc. Brew. Chem.*, 1957, 56–59.—The determination of the oil content of cereal adjuncts is carried out by column extraction. Light petroleum is placed in the extraction column (12 in. × 0·5 in.) to a depth of 4 in. and a 10-g portion of finely ground cereal adjunct is slowly added to the column. The solvent (200 ml) is allowed to flow through the column at the rate of 1 drop per sec. Extraction is continued for about 3 hr. The solvent is distilled off under vacuum and the oil dried. The method works equally well for all the common types of cereal adjuncts and is adaptable to materials other than those referred to as brewing adjuncts. Results agree well with those by the A.S.B.C. method, and replicate results agree within ± 0·02%. K. R. C.

3187. **Protein determinations in cereals using boric acid and the formol titration.** W. Seibel. *Getreide u. Mehl*, 1958, **8** (11), 86–88.—Methods for shortening the time of determination of protein by the Kjeldahl method are discussed. The destruction time is reduced, without loss of N, by the addition of 3 ml of H_2O_2. The titration time is reduced by distilling into a 2% H_3BO_3 soln. and titrating the NH_3 directly with standard acid, with methyl red as indicator. The distillation process is eliminated in the formaldehyde method: formaldehyde soln. is added to the acid digest (after adjustment of the pH to 7·6), producing hexamine and free H_2SO_4, and the acid is titrated potentiometrically. Results agree well with those obtained by the standard method. J. V. RUSSO

3188. **Determination of crude fibre.** A. H. Hallab (Feed and Fertiliser Lab., Louisiana Exp. Sta., Baton Rouge, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (4), 898.—To facilitate filtration, a fibre-glass disc on a Witte plate in a suction funnel is used to support an asbestos mat 5 mm thick. A. A. ELDRIDGE

3189. **Investigation of sitosteryl palmitate as a method for the identification of hard-grain pasta.** L. Laporta (Lab. Chim. Provinc., Pescara, Italy). *Boll. Lab. Chim. Provinciali*, 1958, **9** (1), 100–103.—The absence of sitosteryl palmitate from an acetone extract of flour (Matweef test) gives no clear indication of the use of hard-grain flour, since many such show small quantities of this substance. Fraud would be made more difficult if the flour used in pasta were restricted to those varieties which gave a negative Matweef test. However, no analytical test can detect cases of fraud when soft-grain flour giving a negative Matweef test has been used. J. H. WATON

3190. **Determination of alcohol in fish and egg products.** F. Hillig (Div. Food, Food and Drug Admin., Dept. of Health, Educ. and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (4), 776–781.—The sample is deproteinised with tungstophosphoric acid and the filtrate is distilled with steam after treatment with 2:4-dinitrophenylhydrazine to remove aldehydes and ketones. Alcohol in the distillate is determined iodimetrically after oxidation with $KMnO_4$. The results provide an indication of the presence or absence of spoiled raw material. A. A. ELDRIDGE

3191. **Determination of antibiotic residues in fish.** A. Montefredine, C. Testa and I. Morelli (Lab. Chim. Provinc., Pescara, Italy). *Boll. Lab. Chim. Provinciali*, 1958, **9** (3), 254–262.—A microbiological method is described for determining antibiotic residues of the tetracycline type. The antibiotic is extracted from the sample with water instead of the acidified acetone previously used (*cf.* Grady and Williams, *Anal. Abstr.*, 1954, **1**, 367). The method serves for the quant. determination of residues of antibiotic in the range 0·5 to 2·0 p.p.m., and for the detection of lower quantities. J. H. WATON

3192. **Determination of tetracycline in fish preserved with antibiotics. (Preliminary note.)** A. Montefredine, I. Morelli and C. Testa (Lab. Chim. Provinc., Pescara, Italy). *Boll. Lab. Chim. Provinciali*, 1958, **9** (3), 263–267.—Various Adriatic fish, which show no bacterial inhibition phenomena, have been submitted to microbiological assay after having been washed with water and preserved in ice, both water and ice containing 10 p.p.m. of tetracycline, oxytetracycline or chlortetracycline. After several days, the amount of residual antibiotic in the fish rarely exceeds 1 p.p.m. (a little more when oxytetracycline is used), and this amount is destroyed on cooking the fish in water for 10 to 15 min. J. J. WATON

3193. **Gas-liquid chromatographic method for volatile fatty acids in milk.** C. L. Hankinson, W. J. Harper and E. Mikolajak (Ohio Agric. Exp. Sta., Wooster, U.S.A.). *J. Dairy Sci.*, 1958, **41** (11), 1502–1509.—Chromatography is carried out in a glass tube (6 mm × 4 ft.) packed with Celite 545 containing 25% of silicone oil (D.C. 550) with 5% w/w of stearic acid added. The carrier gas is N (100 cm Hg) and the tube, initially at 60°, is progressively heated in glycerol to 120° during 40 min. and then rapidly to 160° for 80 min. Quant. recovery of seven fatty acids is achieved in the chromatography, but from milk, with the modified Röse-Gottlieb procedure described, the recovery varies from 57% (for formic acid) to 100%. Results are reproducible to within ± 5%. W. H. C. SHAW

3194. **Chromatographic technique for the estimation of some of the free fatty acids in lipolysed dairy products.** A. R. Kemp and J. H. Hetrick (Dean Milk Co., Rockford, Ill., U.S.A.). *J. Dairy Sci.*, 1958, **41** (11), 1494–1501.—The separation of short-chain fatty acids (butyric to capric) from each other and from lauric and higher fatty acids is achieved on silica columns carrying, as stationary phase, methanol containing NH_3 and bromocresol green. Development with *iso*octane saturated with methanol gives fractions which are titrated to phenolphthalein with 0·015 *N* KOH in *iso*propyl alcohol and methanol. Procedures are given for preparing samples for chromatography. Results for lower fatty acids in butter fat agree well with those given by other methods. W. H. C. SHAW

3195. **Milk lipase. Determination of milk lipase activity.** J. Stadhouders and H. Mulder (Lab. Dairying, Agric. Univ., Wageningen). *Ned. Melken Zuiveltijdschr.*, 1958, **12**, 117–129.—Milk was drawn so as to avoid contamination by lipases from micro-organisms and was kept in brown-glass bottles and treated with H_2S to prevent destruction of lipase by light. The substrate tributyrin, homogenised with skimmed milk, was shaken with the milk for a sufficient length of time to ensure stability of the emulsion during incubation at 15° in borax buffer at pH 9·15. No destruction of lipase occurred and the relation between time of incubation and activity was linear. After incubation, 97% extraction of butyric acid was obtained with successive mixtures of ether and light petroleum, and the extract was titrated with 0·1 *N* Na ethoxide. The blank determination was made with sterilised milk in place of raw milk. Lipase activity can be expressed only in arbitrarily chosen units.

NUTR. ABSTR. REV.

3196. **Resazurin disc assay method for detecting antibiotics and natural starter inhibitory activity in milk.** K. M. Shahani and M. C. Badami (Ohio State Univ., Columbus, U.S.A.). *J. Dairy Sci.*, 1958, **41** (11), 1510–1517.—In the method described, 0·5-in. filter-paper discs soaked in the milk sample are placed in contact with an agar medium seeded with a suitable test organism. After incubation at 40° for 40 min. to allow diffusion to take place, the disc is removed. The agar surface is then treated with resazurin soln. and covered with a layer of liquid paraffin. After incubation for up to 2·5 hr., any inhibitory substance originally present in the sample shows up as an area, corresponding to the original position of the disc, where reduction of resazurin is delayed. As little as 0·04 to 0·06 p.p.m. of penicillin, chlortetracycline, oxytetracycline or tetracycline, or 0·11 p.p.m. of streptomycin can be detected. Neither hypochlorite (70 p.p.m.) nor a quaternary ammonium compound (10 p.p.m.) interfered.

W. H. C. SHAW

3197. **Butyric acid value of butter fat.** E. Hanssen (H. Bahlsens Keksfabrik KG, Hanover, Germany). *Dtsch. LebensmittRdsch.*, 1958, **54** (10), 252–254.—Butyric acid values determined by the macro-method of Kuhlmann and Grossfeld (*Z. Untersuch. Lebensmitt.*, 1926, **51**, 31) or by the semi-micro method of Grossfeld (*Z. Untersuch. Lebensmitt.*, 1938, **76**, 340) are reported for about 500 samples in monthly groups covering the period from December 1954 to February 1958, and their significance is discussed. Yearly averages varied only from 19·7 to 20·3, but monthly ones varied from 18·4 to 21·9. Mean values in winter and spring were generally above, and in summer and autumn generally below, the yearly average. The lowest value recorded (in October) was 15·4 and the highest (in January) was 24·5.

E. C. APLING

3198. **Methods for the sampling and chemical analysis of acid casein.** British Standards Institution (2 Park Street, London). B.S. 1417:1955. Amendment No. 1, published 12.3.59.—This contains an amendment to the method of determination of nitrogen. (*Cf. Anal. Abstr.*, 1955, **2**, 1666.)

3199. **Chemistry of non-enzymic browning. IV. Determination of amino acids and amino acid deoxyfructose in browned freeze-dried apricots.** D. L. Ingles and T. M. Reynolds (Div. Food Preserv. and Transport, C.S.I.R.O., Homebush, N.S.W.). *Aust. J. Chem.*, 1958, **11** (4), 575–580.—Free amino acids, ammonia, 1-(N-amino acid)-1-deoxyfructoses (**I**) and related compounds were determined by elution chromatography on columns of a buffered cation-exchange resin by the method described by Moore and Stein (*cf. Anal. Abstr.*, 1955, **2**, 985). Determinations were made before, during, and after storage of samples for 12 months at 25° and 70% r.h. The resolution of proline and glutamic acid proved difficult, but separation was regularly achieved with Zeo-Karb 225–4NC if the column temperature was not raised from 30° to 50° until after the emergence of alanine. The amino acids emerged from the column in the expected order, but the elution vol. were appreciably lower than those recorded by Moore and Stein. Asparagino-deoxyfructose could not be separated from threonino- and serino-deoxyfructose, and valino-deoxyfructose emerged with alanino-deoxyfructose. Ammonia was determined on a 15-cm column run at 50° with 0·4 *N* citrate buffer (pH 5·26). The total free amino acid decreased by 61%, and the ammonia by 9%, the changes in individual amino acids ranging from 22 to 81%. **I** and related compounds accounted for 90% of the free amino acid lost, the actual discrepancy being 1·2 millimoles per 100 g dry wt. of fruit.

K. R. C.

3200. **Determination of chlorophyll and pheophytin in broccoli heated by various procedures.** J. P. Sweeney and M. Martin (Human Nutrition Res. Div., Agric. Res. Service, Dept. Agric., Washington, U.S.A.). *Food Res.*, 1958, **23** (6), 635–647.—The method of Mackinney and Weast (*Ind. Eng. Chem.*, 1940, **32**, 392), based on the quant. conversion of chlorophyll (**I**) into pheophytin (**II**) by treatment with oxalic acid, was modified for a study of the effect of cooking on the chlorophyll content of broccoli. The Comar formula for calculation of the total **I** content of ether extracts was modified for use with acetone extracts. Each extract was examined spectrophotometrically before and after complete conversion of the remaining **I** into **II**. Absorption constants for the change in absorbance were determined at 533, 642·5 and 665 mμ, and from these constants the relative amount of **I** present in the extracts was determined. It was shown that the effect of oxalic acid on the carotene and xanthophylls present was negligible. Chlorophyll *a* (**III**) and *b* (**IV**) were separated by chromatography and the relative stability of the two forms determined, making use of the fact that at 600 mμ their specific absorption constants are the same. It was found that **III** was converted into **II** at a much higher rate than **IV**. The possibility of using Zn to replace Mg eliminated from **I** during the cooking process showed promise although reproducible results have not yet been obtained.

K. R. C.

3201. **Colorimetric determination of micro quantities of vanillin.** G. S. Bains, N. S. Kapur and D. S. Bhatia (Central Food Technol. Res. Inst., Mysore). *J. Sci. Ind. Res., B, India*, 1958, **17** (11), 462–467.—The method described is based on the reaction of vanillin with an excess of thiobarbituric acid in an aq. medium containing specified amounts of HCl or H_3PO_4. The coloured complex shows a single absorption max. at 432 to 434 mμ, and the colour intensity of the complex obeys Beer's law at this wavelength for 1 to 25 μg of vanillin per ml when 2 *N* HCl is the medium used, and for 1 to

40 μg per ml when H_3PO_4 is used. The interference of sugars and the application of the method to processed foodstuffs containing vanillin are discussed. I. JONES

3202. **Separation and identification of artificial colouring matters for use in foodstuffs.** R. De' Gori and F. Grandi (Lab. Chim. Provinc., Siena, Italy). *Boll. Lab. Chim. Provinciali*, 1958, **9** (3), 276–286.—Chromatographic separation and spectrophotometric identification are described for Acid Brilliant Rubine, Cherry red, Amaranth, Ponceau 4R, Ponceau 6R, Salmon red and erythrosin. *Procedure*—The dyes are dissolved in aq. acetic acid, and erythrosin is extracted with diethyl ether. The remaining six dyes are separated into two groups on a circular chromatogram by development with a soln. of 0·20 g of $MgCl_2.6H_2O$ in 15 ml of methanol plus 30 ml of water, Acid Brilliant Rubine and Amaranth constituting the inner zone. Each zone is extracted separately with water and is subjected to ascending chromatography with water - butanol - ethanol (5 : 5 : 4) as solvent. Salmon red, Cherry red, Ponceau 4R and Ponceau 6R are separated on one chromatogram with R_F values of 0·69, 0·59, 0·28 and 0·10, respectively, and Acid Brilliant Rubine and Amaranth with R_F values of 0·41 and 0·12, respectively, on the other. Data are presented for each dye, and extinction curves are given for the range 350 to 570 mμ. J. H. WATON

3203. **Paper chromatography of some natural colours used in food.** R. C. T. Puche (Oficina Química Municipal, Rosario, Sante Fé). *Rev. Asoc. Bioquim. Argentina*, 1958, **23**, 225–227.—R_F values are given for alizarin, turmeric, cochineal, orchil and indigotin in xylene - HCl - H_2O and *n*-butanol - HCl - H_2O solvent mixtures. G. H. FOXLEY

3204. **Partition chromatography on filter-paper of the synthetic colours authorised for use in food. II.** R. C. T. Puche (Oficina Química Municipal, Rosario, Sante Fé). *Rev. Asoc. Bioquim. Argentina*, 1958, **23**, 223–224.—Patent blue and Acid green J cannot be separated with either the xylidine - HCl - H_2O or the *n*-butanol - HCl - H_2O system. Both substances are yellow in these reagents, but give different reactions when dyed on to wool fibres from tartaric acid soln. (For Part I see *Anal. Abstr.*, 1958, **5**, 2793.) G. H. FOXLEY

3205. **Identification by micro-electrophoresis of small quantities of synthetic food colourings.** P. P. Legrand. *Ann. Falsif.*, 1959, **52**, 5–14.—The technique previously described (*cf. Ann. Biol. Clin.*, 1956, No. 12; *Ann. Falsif.*, 1958, **51**, 5) is applied to the identification of synthetic food colours. An aliquot (8 μl) of an aq. soln. of the dye (1 μg per μl, containing Naphthol yellow S as an internal standard) is distributed along a transverse line in the centre of a paper strip, and electrophoresis is continued at pH 8·6 till the Naphthol yellow S band has travelled 5 cm in the direction of the anode (≃ 8 hr.), when the paper is rapidly dried in a current of air and examined in daylight and u.v. light. Most synthetic food colours are electro-negative at pH 8·6, but auramine, malachite green and Paris violet are electropositive. Diagrams are given, and results discussed in detail, for Naphthol yellow S and 26 other synthetic dyes. Mixtures of three or four colours can be separated, even when the mobilities are very similar. When, as for some yellow colours, mobilities are close to those for Naphthol yellow S, a comparison strip is prepared from a soln. of the unknown dye alone, and compared with the main strip. R. E. ESSERY

3206. **Isolation of oil-soluble coal-tar colours from foods.** E. Mark and G. G. McKeown (Food and Drug Lab., Dept. of Nat. Health and Welfare, Ottawa, Canada). *J. Ass. Off. Agric. Chem.*, 1958, **41** (4), 817–818.—Common oil-soluble azo dyes can be removed from maize oil by solvent partition with dimethylformamide and light petroleum. Recoveries of 84·1 to 91·2% are reported. A. A. ELDRIDGE

3207. **Routine method for determination of the activity of baker's yeast.** S. Burrows and J. S. Harrison (Res. and Development Dept., The Distillers Co., Ltd., Epsom, England). *J. Inst. Brew.*, 1959, **65** (1), 39–45.—The volume of CO_2 liberated from a thin dough under standard conditions is measured. Fermentation is carried out in 200-ml bottles immersed in a water bath at 30° ± 0·05° and connected to gas-measuring burettes containing a soln. consisting of 100 g of anhyd. $CaCl_2$ and 5 g of $CuCl_2$ per litre, with a little HCl to ensure complete dissolution (pH > 5·0). The yeast sample (3·57 g) is made up to 100 ml with suspending fluid [27 g of NaCl and 4 g of $(NH_4)_2SO_4$ per litre, fresh daily] and 15 ml of the suspension is transferred to the fermentation bottle. A portion of flour (20 g), which has been pre-heated at 30° for 30 min., is added to the bottle and the whole is stirred for 40 sec. After 13 min., the tap between bottle and burette is opened, and the vol. of gas evolved is measured at atmospheric pressure after 45 min. The burette is then re-set to zero, and the evolved gas read after a further 45 min. Gas vol. are corrected to 760 mm and 20°. The 45-min. vol. corresponds to the old baking-strength test, and the 45 to 90-min. vol. to the proving period. The apparatus and its manipulation are described. Over a 12-month period, average gas-vol. and standard deviations were—45-min. vol., 45 ± 0·6 ml; 45 to 90-min. vol., 55 ± 0·5 ml; total vol., 100 ± 1·0 ml. Random and systematic errors are each about one-third of those of the baking-strength test, giving an 8-fold increase in accuracy. R. E. ESSERY

3208. **Determination of the alkalinity of cocoa ash.** S. Anselmi and L. Boniforti (Ist. Sup. di Sanità, Rome, Italy). *Boll. Lab. Chim. Provinciali*, 1958, **9** (2), 133–142.—*Procedure*—The sample (10 g) is calcined in a platinum dish at 580° to 600°, and an excess of 0·5 *N* H_2SO_4 (≃ 25 ml) is added. The soln. is kept on a boiling-water bath for 20 min., and is then filtered. Two drops of 0·4% bromocresol green soln. are added, and the soln. is titrated with 0·25 *N* NaOH. The phosphate present is pptd. by the addition of 15 ml of ≃ *N* $AgNO_3$, and 2 drops of methyl red are added. The soln. is titrated again with 0·25 *N* NaOH until the colour changes from yellow - orange to yellow - green; the addition of alkali must be very slow near the end-point. The first back-titration gives the alkalinity in the ash due to oxides, carbonates and phosphates, and the total back-titration that due to oxides and carbonates. The method is suitable for the determination of alkali in any vegetable ash or other product where the residue contains phosphate. J. H. WATON

3209. **Determination of the amino nitrogen in orange juices and orangeades by the formol method.** A. Chindemi and A. Cultrera (Lab. Chim. Provinc., Terni, Italy). *Boll. Lab. Chim. Provinciali*, 1958, **9** (3), 311–322.—It is confirmed that methods for the determination of the formol number can only lead to approx. results, which are, however, accurate enough for the assessment of the genuineness of the article. Electrometric modifications of existing methods are described. J. H. WATON

3210. **Experience with the conductimetric determination of α-acids of hops with respect to brewing value. Criticism of a paper of the above title by B. Trolle and R. Djurtoft.** B. D. Hartong, H. E. Jansen and P. Mendlik (Phoenix Brewery, Rotterdam, Holland). *Brauwissenschaft*, 1959, **12** (2), 43.—The statement in the paper referred to (*Ibid.*, 1958, **11**, 283) that $CHCl_3$ is a better extraction medium for the α-acids than benzene is incorrect; data are given showing that the two solvents are of equal value. Objection is also taken to the statement that the α-acids constitute "about" 50% of the total acids: the actual variations are from 45 to 75%. P. S. ARUP

3211. **Rapid paper-chromatographic determination of carbohydrates in worts or beers. (Observation of decomposition of carbohydrates during fermentation.)** H. R. Stocker. *Schweiz. Brauerei Rdsch.*, 1958, **69** (10), 167–168.—The sugars (including the polymaltoses) contained in the samples (filtered if in the course of fermentation) can be separated by development (in closed vessels) for 12 to 15 hr. with aq. 77% *n*-propanol as ascending solvent, on paper sheets from which lozenge-shaped excisions have been made near the base to form "bridges" 2 cm wide on which the test samples (0·015 to 0·25 ml) are deposited. The spraying reagent consists of a 2% soln. of diphenylamine, aniline in ethanol, and 85% H_3PO_4 (5:5:1). The spots are located and evaluated by comparison with spots obtained from parallel runs with standard soln. of the individual sugars. P. S. ARUP

3212. **Determination of dextrin in wort and beer. Criticism of the conventional method and development of a new procedure.** E. Schild and H. Weyh (Tech. Hochschule, Munich, Germany). *Brauwissenschaft*, 1958, **11** (9), 198–204; (10), 232–235; (11), 255–262.—Sources of error inherent in the conventional method (acid hydrolysis and determination of dextrin as reducing sugar) are examined with a view to accounting for the deficit of $\simeq 15\%$ between the actual extract and the sum of the separately determined constituents. Several analytical modifications are tested. The recommended method gives values for dextrin which reduce the deficit to $< 1\%$. *Procedure*—To remove all carbohydrates except dextrin and hexosans, ferment the sample (150 ml) with fresh brewer's yeast (3 g) for 3 to 5 days at 25°. Filter, remove the CO_2, dilute the filtrate (110 g) with water (50 ml), distil off 100 to 120 ml, and dilute the residue to 110 g. To the diluted residue (100 g) add water (100 ml) and 25% HCl (20 ml), heat under reflux for 3 hr. in boiling water, cool, neutralise with NaOH, make up to 500 ml and filter. To 200 ml of the filtrate add yeast (4 g), ferment for 3 to 5 days at 25° and filter. To 75 g of the filtrate (free from CO_2) add water (30 ml) and distil over 70 ml. Dilute the distillate to 75 g with water and determine its ethanol content by sp. gr. measurement. Dextrin % (w/w) in the sample = ethanol % (w/w) × 9. P. S. ARUP

3213. **Separation, characterisation and estimation of polysaccharides in beer.** H. R. Held and W. D. McFarlane (Res. Div., Canadian Breweries Ltd., Toronto). *Proc. Amer. Soc. Brew. Chem.*, 1957, 116–120.—After pptn. of the polysaccharides with acetone, and dissolution of the ppt. in 0·5% $Na_2B_4O_7$ soln. (**I**), paper strips were dipped in **I** and electrophoresis of the polysaccharide soln. was carried out with **I** as buffer at a potential of 300 V for 16 hr. The air-dried guide strips were hung in a fume hood, sprayed with diphenylamine - aniline - H_3PO_4 soln. and heated at 90° for 10 min. The dextrins, which showed up as a blue band, had moved about 1 in. towards the cathode, and the pentosans, which appeared as a light-brown band, had moved ahead of the dextrins and were located about 2 in. from the point of application. Dextrins were determined by anthrone, and pentosans with Bial's orcinol reagent. With monosaccharides, electrophoresis was carried out for 3 hr. only. All glucosans moved to the same position as maltose. Apparently pentosans and dextrins are separated on paper chromatograms by moving with the electro-osmotic flow, being retarded to a different degree by adsorption on the paper. The araboxylan content of beer comprises 46% of the total pentosans in beer. Evidence is presented which indicates that the pentosan gums, although present in small amounts, account for 30% of the viscosity of beer. K. R. C.

3214. **Determination of tannins in beer and brewing materials by ultra-violet spectrophotometry.** J. L. Owades, G. Rubin and M. W. Brenner (Schwarz Lab., Inc., Mt. Vernon, New York). *Proc. Amer. Soc. Brew. Chem.*, 1958, 66–73.—The *iso*humulones are first removed from the wort or beer by extraction with *iso*octane. The tannins are then extracted with ethyl acetate, and, after evaporation of the solvent, are dissolved in acidified methanol. The absorbance of the methanolic soln. is measured at 270 mμ. The method previously described (*J. Agric. Food Chem.*, 1958, **6**, 44) is modified; both the sample size and the amount of *N* HCl are increased to 5 ml and the acidified sample is now extracted with one 50-ml portion of ethyl acetate instead of with three small portions. The level of tannins at each stage of the brewing process is determined. The present method gains greatly in accuracy by using as the standard the actual materials being determined, the standard absorbance curve for these being based on the tannins isolated from four malts and three hops. Reproducibility data gave a standard deviation of 3·35 p.p.m. K. R. C.

3215. **Determination and detection of higher alcohols in fermented liquids by gas-phase chromatography.** L. Fleischmann and A. Daghetta (Centr. di Stud. Chim. del. Ferment., Milan). *Ric. Sci.*, 1958, **28** (11), 2286–2290.—A gas-chromatographic procedure has been developed for the determination of higher alcohols in beer and wine, after preliminary distillation and dehydration. The only detectable higher alcohols in a range of Italian products were *iso*butyl and *iso*amyl alcohols. These were present in the ethanol to the extent of 0·12 to 0·18% and 0·25 to 0·32%, respectively, for the beers and wines. A colorimetric determination gave similar results. L. A. O'NEILL

3216. **Total sulphydryl content of beer. II. Changes during brewing.** M. W. Brenner, G. J. Schapiro and J. L. Owades (Schwarz Lab., Inc., Mt. Vernon, New York). *Proc. Amer. Soc. Brew. Chem.*, 1958, 104–111.—The determination is based on the amperometric titration of –SH groups with Ag^+. Glutathione is used to arrest the reaction of an initial excess of $AgNO_3$ at known time intervals, and the amount of –SH reacting in "zero time" is determined graphically. The present method is a modification of that previously presented (*cf.* Brenner *et al.*, *Anal. Abstr.*, 1958, **5**, 2396). Further experience of the method confirmed that the difference between a 1-min. titration and the "zero time" value was less than 5%, and the time required for routine testing was therefore reduced by substituting a 1-min. titration for the graphical derivation of "zero time" from the 2-, 4- and 6-min. titrations. The total –SH level of 14 worts ranged from 0·2 p.p.m. to 1·1 p.p.m. Increases in total –SH level occurred during fermentation in seven of nine pilot-plant brews examined. Exposure of beer to light caused an increase in the total –SH content. K. R. C.

3217. **Determination of total volatile oxidisable matter in fermentation gas.** H. L. Ziliotto, R. C. Turczany and J. B. Bockelmann (F. M. Schaefer Brewing Co., Brooklyn, New York). *Proc. Amer. Soc. Brew. Chem.*, 1958, 75–79.—Impurities are absorbed from the gas by its passage through two absorbers in series; the first (containing 0·25 *N* $K_2Cr_2O_7$ and Ag_2SO_4 in conc. H_2SO_4) removes organic materials and H_2S; the second (dil. $K_2Cr_2O_7$ soln.) traps any SO_2 escaping from the first. Refluxing the first absorbant with H_2SO_4 (1:1) and 0·25 *N* $K_2Cr_2O_7$, followed by titration against $(NH_4)_2SO_4.FeSO_4.6H_2O$ soln., with 1:10-phenanthroline - ferrous complex as indicator, yielded good recoveries of many compounds of types probably present in fermentation gas. Analyses at different stages of fermentation showed that the oxidisable matter in the CO_2 continues to increase as fermentation progresses. In a CO_2 purification plant the complete process removes 95% of the oxygen demand of the incoming gas. By this method it was possible to evaluate the processes used for the purification of CO_2 produced by fermentation. The information thus obtained led to improvements in the design of new purification equipment. K. R. C.

3218. **Sensitive direct colorimetric technique for the determination of sulphur dioxide in beer.** I. Stone and C. Laschiver (Wallerstein Lab., New York, U.S.A.). *Proc. Amer. Soc. Brew. Chem.*, 1957, 46–55.—The colour produced with acid-decolorised pararosaniline hydrochloride (**I**) and formaldehyde (**II**) is measured photometrically, the beer being treated directly, without distillation. *Procedure for free SO_2*—Dilute 10 ml of beer to 100 ml. To 25 ml of the dilution add 5 ml of **I** soln. [1 g of **I** dissolved in 200 ml of H_2O, treated with 40 ml of HCl (1:1), made up to 250 ml and allowed to stand for 15 min.], followed by 5 ml of **II** soln. (5 ml of 40% **II** made up to 1 litre). Make up to 50 ml, maintain at 25° for 30 min. and read the extinction at 550 mμ (or with a green filter). Refer results to a calibration curve. *Total SO_2*—Transfer 10 ml of beer to a flask containing 2 ml of $HgCl_2$ soln. (**III**) (27·2 g of $HgCl_2$ and 11·7 g of NaCl in 8 litres of H_2O) and 5 ml of 0·1 *N* H_2SO_4. Mix, add 15 ml of 0·1 *N* NaOH, mix, and set aside for 15 sec., then add 10 ml of 0·1 *N* H_2SO_4 and mix. Make up to 100 ml and take 25 ml for colour development. *Blank*—To 10 ml of beer add 0·5 ml of 1% soluble starch soln., then 0·05 *N* iodine dropwise till a permanent pale-blue colour is formed, and 1 drop in excess. Dilute to 100 ml, set aside till the blue colour fades, then treat 25 ml for colour development as before. *Calibration curve*—To 100 ml of **III** add 1 ml of a standard soln. of $NaHSO_3$ (≡ 10 mg of SO_2). Dilute to 500 ml, add aliquots containing up to 160 μg of SO_2 to 10-ml aliquots of a beer sample. Make each up to 100 ml, and develop and read the colours as before. Results are highly reproducible, and agree well with those by the Monier-Williams method. Recoveries of SO_2 added to beer were satisfactory. K. R. C.

3219. **Residue determination of Sevin (1-naphthyl methylcarbamate) in wine by cholinesterase inhibition and paper chromatography.** G. Zweig and T. E. Archer (Pesticide Residue Res. Lab., Univ. of California, Davis). *J. Agric. Food Chem.*, 1958, **6** (12), 910–913.—The insecticide Sevin is extracted from wine with $CHCl_3$ - ethanol (1:1); the residue, after removal of solvents, is dissolved in 50% aq. methanol, extracted with light petroleum to remove waxes, and extracted into redistilled benzene, which is dried over Na_2SO_4, and evaporated under vacuum. The residue is dissolved in benzene for the determination of Sevin by means of horse-plasma cholinesterase, or in methanol for the determination of both Sevin and its breakdown product (1-naphthol) by paper chromatography: the paper is sprayed with 1·5 *N* methanolic KOH - *n*-butanol (1:1), which hydrolyses Sevin to 1-naphthol, and after drying it is dipped into 0·01% *p*-nitrophenyldiazonium fluoroborate dissolved in methanol - ether (1:1), giving blue spots. R_F values were 0·37 for 1-naphthol, and 0·65 for Sevin. M. D. Anderson

3220. **Micro-determination of the saponification number.** F. A. Lee (N.Y. State Agric. Exp. Sta., Cornell Univ., Geneva, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (4), 899–900.—The sample of oil (40 mg) with 0·5 ml of ethanolic KOH soln. (4%), contained in a micro-Kjeldahl flask, is maintained at 75° for 50 min. and subsequently titrated. A blank is also run. A. A. Eldridge

3221. **A new procedure for the determination of volatile carbonylic substances in autoxidising edible fats.** C. H. Lea and P. A. T. Swoboda (Low Temp. Res. Sta., Univ., Cambridge, England). *Chem. & Ind.*, 1958, (31), 974–975.—The method of Chang and Kummerow (*J. Amer. Oil Chem. Soc.*, 1955, **32**, 341) for the determination of volatile carbonylic compounds gives results ≃ 5% of those attainable by the separation of the volatile constituents from oxidised fats by distillation for 0·5 hr. at 50° and 0·01 mm (Hg). The conditions for obtaining max. recovery are discussed. H. B. Heath

3222. **Spectrophotofluorimetry of edible oils.** F. De Francesco (Lab. Chim. Provinc., Trento, Italy). *Boll. Lab. Chim. Provinciali*, 1958, **9** (1), 76–89.—Pure olive oil, rectified and synthetic olive oils and their mixtures are examined spectrofluorimetrically. The apparatus, which is fitted with a Wood's lamp, covers the range from 400 to 1000 mμ. Fluorescence curves are drawn for uncorrected readings, the instrument being standardised with a 0·01% quinine acid sulphate soln. in 0·1 *N* H_2SO_4. Pure

olive oil is revealed unambiguously by its fluorescence spectrum, and information is obtained on the nature of other commercial olive oils and mixtures. J. H. WATON

3223. **Spectrophotometric examination of olive oils in the visible region.** A. Fabbietti (Lab. Chim. Provinc., Rovigo, Italy). *Boll. Lab. Chim. Provinciali*, 1958, **9** (3), 287–296.—Samples of olive oils are submitted to the Liebermann reaction with $CHCl_3$ and an H_2SO_4 - acetic anhydride mixture, and the extinction of the product is measured at eight different wavelengths. From relationships between these values, information as to the type and genuineness of the oils may be deduced. J. H. WATON

3224. **Identification of rapeseed oil in olive oil by urea fractionation.** V. R. Bhalerao and J. H. Mahon (Canada Dept. Nat. Health and Welfare, Food and Drug Lab., Ottawa). *J. Ass. Off. Agric. Chem.*, 1958, **41** (4), 745–749.—Rapeseed oil contains 35 to 55% of erucic acid, which is absent from olive oil. Long-chain fatty acids form adducts with urea in methanol more readily than do short-chain fatty acids. The oil is saponified and the fatty acids (8 g) are mixed with urea (30 g) in methanol (200 ml); the mixture is kept at 25° for 1 hr., the acids being then recovered from the ppt. by heating with water. The procedure is repeated with these recovered acids, and the saponification value and mol. wt. are determined on the final product. Olive oil that yields fatty acids having a mol. wt. > 280 is considered to be adulterated with an oil containing erucic acid glycerides. A. A. ELDRIDGE

3225. **Determination of sesamin in sesame oil.** A. Chindemi (Lab. Chim. Provinc., Terni, Italy). *Boll. Lab. Chim. Provinciali*, 1958, **9** (3), 297–310.—The spectrophotometric determination of sesamin in sesame oil is based on the colour reaction described by Pavolini and Isidoro (*Olii Min.*, 1952, **29**, 33). *Procedure*—The sample (0·5 g) is dissolved in 250 ml of benzene, and a 5-ml aliquot is introduced into a 25-ml flask. To this are added 5 drops of acetic anhydride, 3 to 4 drops of a 2% ethanolic soln. of furfuraldehyde and exactly 0·2 ml of conc. H_2SO_4, added dropwise and with stirring, when a brilliant green colour develops. After 15 min., the soln. is diluted to 25 ml with benzene and the extinction is read against a blank in a 1-cm cell at 620 mμ. J. H. WATON

3226. **Artificially coloured foodstuffs. Identification and differentiation of butter yellow and aniline yellow in oil and in edible fats.** L. Polzella (Lab. Chim. Municip., Rome, Italy). *Boll. Lab. Chim. Provinciali*, 1958, **9** (1), 96–99.—Aniline yellow (*p*-aminoazobenzene) and butter yellow (*p*-dimethylaminoazobenzene) are distinguished from each other by the formation of Lauth's violet (thionine) and methylene blue. *Procedure*—The sample ($\simeq$ 20 ml of oil or a similar quantity of a solid fat) is dissolved in 40 ml of diethyl ether and reduced with 3 ml of $SnCl_2$ soln. (10% in conc. HCl). The soln. is shaken with 25 ml of H_2O, and the aq. layer is transferred to a second separating-funnel where, after being neutralised with 20% NaOH soln. to phenolphthalein, it is shaken with $CHCl_3$ (2 × 10 ml). The $CHCl_3$ extracts are combined, filtered and evaporated to $\simeq$ 3 ml. The soln. is transferred to a test-tube and is shaken with an equal vol. of $\simeq$ 0·5 *N* HCl. Then a few drops of a freshly prepared saturated aq. soln. of H_2S are added, followed after further shaking by a drop of 10% $FeCl_3$ soln. The formation of a violet colour indicates the presence of aniline yellow, and a blue colour the presence of butter yellow. J. H. WATON

3227. **Chromatographic separation and determination of thiamine and thiamine phosphoric esters on ion-exchange resins.** L. De Giuseppe and G. Rindi (Inst. Human Physiol., Univ. Pavia, Italy). *J. Chromatography*, 1958, **1** (6), 545–551.—A mixture of thiamine and its phosphoric esters was percolated through two superimposed columns containing, respectively, the acetate and borate forms of a strong anion-exchange resin (Dowex IX-8). After addition of an aq. soln. of the sample to the upper (acetate) column and elution with 0·03 *M* H_3BO_3, thiamine pyro- and tri-phosphoric esters were retained by the acetate column and thiamine and the orthophosphoric ester by the borate column. The borate column was separately eluted with 0·1 *M* NaCl in 0·1 *M* HCl, and the acetate column with 0·2 *M* Na acetate in 0·04 *M* acetic acid followed by 0·1 *M* HCl. The components were determined by u.v. spectrophotometry. Recoveries ranged from 91 to 97%. G. P. COOK

3228. **Microbiological determination of vitamin B_6 in wheaten flour, and in fractions of the wheat grain.** K. M. Clegg and J. J. C. Hinton (Res. Ass. of British Flour-Millers, Cereals Res. Sta., St. Albans). *J. Sci. Food Agric.*, 1958, **9** (11), 717–721.—Pyridoxine is determined microbiologically with the yeast *Saccharomyces carlsbergensis* 4228; pyridoxine is used as an internal standard. A medium enriched in thiamine, and of pH 4·5, gives a good response for all three forms of pyridoxine. The method is suitable for small quantities of material. P. D. PARR-RICHARD

3229. **New colour reaction for pyridoxal based upon its interaction with thiophen.** V. E. Levine and W. G. Hansen (Creighton Univ. Med. Sch., Omaha, Neb., U.S.A.). *Biochim. Biophys. Acta*, 1959, **31** (1), 248–249.—To 1 litre of absolute ethanol add 100 mg of phenylhydrazine hydrochloride and 10 ml of conc. H_2SO_4. Allow to stand for 1 week then distil the ethanol. *Procedure*—To 1 ml of test soln. at 0° add 1 ml of 0·5% redistilled thiophen in purified ethanol and 8 ml of 80% (v/v) H_2SO_4, both previously cooled to 0°. Allow the mixture to reach room temp., then heat at 75° to develop max. colour. The extinction at 615 mμ is proportional to concentration in the range 1 to 100 μg per ml. Many short-chain aliphatic ketones and aldehydes give other colours with thiophen, but other compounds closely related to pyridoxine do not interfere. W. H. C. SHAW

3230. **Dead-stop titration method for the determination of ascorbic acid.** J. A. C. van Pinxteren and E. Verloop (Univ., Utrecht, Netherlands). *Pharm. Weekbl.*, 1958, **93** (22), 982–987.—Ascorbic acid in a soln. made slightly alkaline with $NaHCO_3$ can be titrated directly with $K_3Fe(CN)_6$. The end-point is indicated by the deflection of a galvanometer connected to platinum electrodes carrying a current of 50 mV. The titration can be checked by adding a slight excess of $K_3Fe(CN)_6$ and titrating the $K_4Fe(CN)_6$ with $ZnSO_4$ soln. until the galvanometer returns to zero. The method is

suitable for the determination of ascorbic acid in orange juice. Glucose and fructose do not interfere. G. BURGER

3231. **Polarographic determination of vitamin C in vegetables from stimulated and non-stimulated seeds.** M. Sherban and D. Motset-Grigorash (Inst. Biochem., Acad. R.P.R., Bucharest). *Rev. Chim., Acad. R.P.R.*, 1958, **3** (2), 275–279.—*Procedure*—Shake the sample with 1·5% HPO_3 soln. and then grind well. Filter and add a measured aliquot of the extract to an equal vol. of buffer soln. [0·066 *M* KH_2PO_4 - 0·066 *M* Na_2HPO_4 (1:19)]. Polarograph immediately and calculate the ascorbic acid concn. by reference to standard curves obtained with the pure vitamin in HPO_3. K. R. C.

3232. **Cockroaches and their fragments as food contaminants.** H. P. Eiduson, D. Firestone and J. E. Roe (Food and Drug Admin., Dept. of Health, Educ. and Welfare, Chicago, Ill., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (4), 886–898.—Characteristic structures of fragments of cockroaches likely to contaminate food products are described and illustrated. A. A. ELDRIDGE

3233. **Identification of stored-products insects by the micromorphology of the exoskeleton. IX. Head, thorax and abdomen.** H. C. Van Dame, G. L. Reed and P. T. Ikari (Food and Drug Admin., Buffalo, N.Y., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (4), 828–886.—The characteristic appearance of fragments of the head, thorax and abdomen of various insects that infest cereal grains and grain products is described and illustrated.
A. A. ELDRIDGE

See also Abstracts—**2825**, Food analysis (review). **2869**, Separation of ^{137}Cs in foods. **2909**, Determination of Pb in organic matter. **2924**, Determination of P. **3022**, Determination of fructose. **3177**, Determination of sucrose in syrup.

Sanitation

Analysis of air, water, sewage, industrial wastes, industrial poisons.

3234. **Determination of acetylene in air in concentrations from ten parts per thousand million to ten parts per million.** E. E. Hughes and R. Gorden, jun. (National Bureau of Standards, Washington, D.C.). *Anal. Chem.*, 1959, **31** (1), 94–98.—Pass the gas stream at a rate of 50 to 200 ml per min. through a U-tube at −78°, to trap water and the higher alkynes, and then adsorb the acetylene on a column of purified silica gel (1 cm × 0·2 cm) at −78°. Allow the gel to warm to room temp. and treat it with an ammoniacal soln. of cuprous chloride. The quantity of acetylene is indicated by the intensity of the blue colour produced in the gel. Quant. results are easily obtained in the range from 0·1 to 10 p.p.m., and concn. as low as 0·001 p.p.m. may be determined. Mercaptans, H_2S, propyne, butyne and pentyne interfere.
A. R. ROGERS

3235. **Analysis of solvents injurious to health in technical mixtures. Comparative consideration of the application of infra-red spectroscopy and gas chromatography.** E. G. Hoffmann (Max-Planck-Inst. f. Kohlenforsch., Mülheim-Ruhr, Germany). *Z. anal. Chem.*, 1958, **164** (1), 182–218.—A critical study of i.r. spectroscopic and gas-chromatographic methods is made for various solvents, such as aromatic compounds with b.p. up to 150°, and methanol, CS_2 and organic halogen compounds with b.p. up to 200°. The elimination of difficulties caused by superimpositions of characteristic absorptions and peaks, and by displacement of absorption frequencies, etc., is discussed. Extinction coeff. of 20 analytical bands depending on the extinction and nominal resolving power of the spectrometer are shown. Sensitivity factors for the rectification of area are calculated from kinetic data, and the results are tabulated. B. B. BAUMINGER

3236. **Improvements in or relating to the detection of mercury vapour in air or other gas.** O. H. Drager. Brit. Pat. 811,408, date appl. 2.4.57. Germany, date appl. 17.4.56.—The gas is passed through silica gel impregnated with $HgBr_2$ and $AuBr_3$. Any Hg in the gas reduces the $HgBr_2$ to Hg_2Br_2 which then reduces the $AuBr_3$ to colloidal Au, whereby the colour of the reagent changes from yellow to intense red - violet.
J. M. JACOBS

3237. **An electrode system for measuring dissolved oxygen.** D. E. Carritt and J. W. Kanwisher (The Johns Hopkins Univ., Baltimore, Md., U.S.A.). *Anal. Chem.*, 1959, **31** (1), 5–9.—A polyethylene-membrane covering on the electrodes confers selectivity for dissolved oxygen on the system and so overcomes the major disadvantages of existing polarographic methods for determining dissolved oxygen. The system, which is temp. compensated, functions satisfactorily in a wide variety of aq. media and can be used with continuously recording equipment. The long-term zero drift is negligible.
K. A. PROCTOR

3238. **Automatic potentiometric titration in the determination of dissolved oxygen.** V. S. Griffiths and M. I. Jackman (Chem. Dept., Battersea College of Technol., London, England). *Anal. Chem.*, 1959, **31** (1), 161–162.—The titration of dissolved O in the range 0·1 to 8 p.p.m. was followed potentiometrically by using an electrode pair, comprising a glass reference electrode and a platinum indicator electrode. The average deviation from the mean by this method was 1·85% compared with 3·94% by the Winkler method. K. A. PROCTOR

3239. **Dissolved oxygen determination.** I. Nusbaum (Calif. Water Pollution Control Board No. 9, San Diego). *Wat. & Sewage Wks*, 1958, **105** (11), 469–471.—This method is exactly similar to the standard Winkler method for dissolved oxygen determination, but sulphamic acid (4 g for a 250-ml sample) is used instead of conc. H_2SO_4. Results agree with those obtained by the standard Winkler method. S.C.I. ABSTR.

3240. **Manometric method for the rapid practical determination of biochemical oxygen demand.** E. O. Dillingham, D. T. Knuth and G. E. Wessman. *TAPPI*, 1958, **41**, 321–333.—A pure-culture manometric technique has been developed as a rapid method of predicting the B.O.D. of waste waters from pulp and paper mills. The method depends on massive inoculation of the sample with a standardised suspension of *Aerobacter aerogenes* of high physiological activity and high reaction temp. (37°). A comparison was made between the manometric B.O.D. and the 5-day B.O.D. for spent

sulphite liquor, spent semi-chemical liquor, de-inking waste waters, kraft pulp mill waste waters, kraft white liquor, and river water, and the relation between the two appeared to be adequate for the prediction of the 5-day B.O.D. from the manometric B.O.D. Although the manometric method is limited in some respects and is more expensive to use than the 5-day dilution method, it is considered to be practical for the routine control of day-to-day disposal problems.

WAT. POLLUT. ABSTR.

3241. **Flame photometry of lithium in natural water.** Yasumitsu Uzumasa, Yoshikazu Nasu and Toshiko Seo (Fac. of Sci., Hokkaido Univ., Sapporo). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1958, **79** (11), 1292–1295.—In order to decrease the time taken for the separation of Li (1 to 10 mg per litre) from other alkali metals in natural water, its determination by flame photometry (H, 4·0 lb, O, 14 lb per sq. in.) was examined with reference to the elimination of the interference due to Na, Ca, Mg and SO_4^{2-}. With increase in Na^+ concn., the intensity of the Li line at 671 mμ decreases for <1 g of Na per litre and increases for >2 g of Na. The effect of Mg and Ca is masked by Al^{3+} (0·03 to 0·05 *M*). The amount of the sample is so chosen that the concn. of Na in the final soln. is 2 g per litre. When the amounts of Ca and Mg are >500 and >200 mg per litre of the final soln., respectively, they are removed as oxalate and oxinate; Fe, Al and SO_4^{2-} are removed with aq. NH_3 and $BaCl_2$. The filtrate is evaporated to dryness, the residue is heated to decompose organic substances and the excess of NH_4Cl, then dissolved in a small amount of water, filtered to remove SiO_2, and mixed with $Al_2(SO_4)_3$ to produce a 0·03 *M* soln.; the pH is adjusted to 1 to 3·2, the soln. made up to 100 (or 250) ml and submitted to flame photometry. The mean of the blank values at 640 and 700 mμ is taken as the blank value at 671 mμ.

K. SAITO

3242. **Determination of sodium and potassium in water by flame photometry. (Preliminary note.)** M. Mengoli and A. Boari (Lab. Chim. Provinc., Bologna, Italy). *Boll. Lab. Chim. Provinciali*, 1958, **9** (3), 341–354.—Potassium in water is determined with the aid of a flame photometer by the "method of increments". No internal standard is necessary, and the method is a direct reading one. *Procedure*—The sample (1 litre) is reduced in vol. to 100 ml, which is divided into five equal parts. The K in one part is estimated approx. by the flame photometer without any correction for the other elements present. Then, to the other four portions are added 0, 1/4, 1/2 and 1/1 of this amount of K as KCl soln. (≥ 1 ml) by means of a micro-burette. After diluting to equal vol. with water, the soln. are measured in the flame photometer, taking the reading for the most concentrated soln. as 100, and that for distilled water as zero. A graph is plotted of galvanometer deviation *vs.* concn. of K added. The graph is almost linear and, on extrapolating to zero galvanometer deviation, gives the value of K in the sample as the intercept between this point and the origin. For concn. of K between 5 and 20 μg per ml, the K lines at 765 and 770 mμ are used together. Whilst the results are less accurate than those obtained by normal spectrochemical analysis, much less time is required.

J. H. WATON

3243. **Spectrophotometric determination of fluorine with particular reference to water.** B. Visintin and S. Monteriolo. *R. C. Ist. Sup. Sanit.*, 1958, **21**, 338–417.—After a detailed review of colorimetric and spectrophotometric methods for the determination of low concn. of F, experiments are reported on the application of three of the methods to the determination of F in water. The pertitanic acid method of Monnier *et al.* (*Helv. Chim. Acta*, 1950, **33**, 1) was not sufficiently sensitive and the results were not reproducible. A spectrophotometric technique in which the Zr - alizarin reagent was used (Casares, J. and Casares, R., *An. Soc. Esp. Fis. Quim.*, 1930, **28**, 1159) was found to be the most suitable for the analysis of drinking water. Fluoride ions in concn. up to 1·5 mg per litre could be determined by direct reading at 530 mμ with an accuracy of 0·03 mg per litre and concn. between 1·5 and 2·0 mg per litre with an accuracy of 0·05 mg per litre. The most suitable reagent was obtained with concn. of 8×10^{-4} *M* alizarin and 4×10^{-4} *M* zirconium in 1·35 *N* H_2SO_4. Factors affecting the sensitivity and stability of the reagent, and the chromatic reaction, were also investigated with a view to correcting the photometric readings. The aluminium - Eriochrome cyanine method (Thrun, *Anal. Chem.*, 1950, **22**, 918) was found to be the most suitable method for the analysis of water containing high concn. of minerals. The F^- can be determined in concn. of 2 to 8 mg per litre with a reproducibility of $\pm$ 0·05 mg per litre, provided that the temp. and the duration of the reading do not differ by more than $\pm$ 1° and 5 min., respectively, from the values used in the construction of the calibration curve.

WAT. POLLUT. ABSTR.

3244. **Determination of radioactive iodine in water and sewage.** R. B. Hahn, S. Levin and R. L. Friedlander (Dept. Chem., Wayne State Univ., Detroit, Mich.). *J. Amer. Water Wks Ass.*, 1958, **50** (11), 1499–1504.—The radioactive iodine (**I**) in drinking water is pptd. as AgI after adding KI as carrier and excess of Cl^-. After removal of the AgCl by treatment of the ppt. with aq. NH_3, the resulting AgI is dissolved by addition of zinc and H_2SO_4. The **I** is then separated from other fission products by the method of Glendenin and Metcalf (*cf.* "Radiochemical Studies; Book 3: The Fission Products," McGraw Hill Book Co. Inc., New York, 1951, p. 1625). **I** in sewage may exist partly as organically bound iodine, so a radioactive iodine tracer, 80% in the form of iodo-casein and 20% in ionic form, is added to representative samples of sewage effluent. Sodium hypochlorite in alkaline soln. is used to completely decompose the organic matter and to convert the iodine quantitatively into the ionic form. Results by both procedures are presented and discussed.

O. M. WHITTON

3245. **Determination of synthetic detergent content of raw-water supplies.** American Water Works Ass., Task Group 2266P. *J. Amer. Wat. Wks Ass.*, 1958, **50** (10), 1343–1352.—In this report, a review is presented, with detailed descriptions of the methylene blue and i.r. processes for the determination of alkylarylsulphonate content. The i.r. process, involving isolation from the detergent of a few milligrams of the surface-active agent (the most widely used is the alkylbenzenesulphonate derived from polymerised propylene) and i.r. spectrophotometry of an amine complex, is long and complicated, but is accurate for low concn. The methylene blue process, depending on the

formation of a blue salt between methylene blue and alkylarylsulphonate, followed by spectrophotometry at 652 mμ, is subject to interference. (9 references.) S.C.I. Abstr.

3246. **Comparison of the 2-naphthol and molybdenum reagents for the detection of sucrose in waters.** M. Friml. *Listy Cukr.*, 1958, **74** (12), 277–278.—It has been stated that the reaction based on the inversion of sucrose with H_2SO_4 and detection of the resulting (hydroxymethyl)-furaldehyde with 2-naphthol is more sensitive than that with molybdenum reagent (ammonium molybdate in H_2SO_4). Neither reaction is specific for sucrose. Interference is caused by Fe^{3+}, but not by 0·1% of NH_3, NH_4Cl, Na_2CO_3, Na_2HPO_4 or Na_2SO_3. J. Zýka

3247. **Determination of radioactivity in ground and surface waters.** E. Wosahlo. *Gas- u. Wasserfach*, 1958, **99**, 322–324.—In connection with the drawing up of standard methods, a method is described for determining radioactivity and the radioactive elements present by dividing the residue on evaporation of the water with HCl into four fractions. Of these the first is matter insol. in HCl, the second contains Ca, Sr and Ba as sulphate, the third contains the K as potassium aluminium sulphate, and the fourth contains Mg and residual substances. The method of fractionation is described and an account is given of its use in the examination of various ground waters and a surface water. Activities, based on β-radiation, below 10^{-7} μC per ml were determined with sufficient accuracy. The advantages of the method and the importance of the detection of natural and artificially added radioactive substances are discussed. Wat. Pollut. Abstr.

3248. **Determination of radio-strontium in sea water.** B. A. Loveridge (A.E.R.E., Harwell, Berks., England). A.E.R.E. Report C/M 380, 1959, 7 pp.—Up to one litre of filtered sea water may be analysed by a procedure similar to one already described (*cf.* Loveridge and Thomas, *Anal. Abstr.*, 1958, **5**, 1389). Strontium carrier (≡ 40 mg of Sr) is added to the sample and after heating for 15 min. Sr is pptd. as the oxalate. The ppt. is separated and dissolved, and the Sr is then pptd. four times as $Sr(NO_3)_2$ and finally, after scavenging with $Fe(OH)_3$ and $BaCrO_4$, as Sr oxalate, which is β-counted and compared with a standard source containing ^{90}Sr. The natural content of Sr in sea water is taken as 8 p.p.m. The recoveries of ^{90}Sr in 16 experiments were all in the range 102 ± 4%. G. J. Hunter

3249. **Radiochemical separation of fission products from large volumes of sea water. Strontium, caesium, cerium and promethium.** T. T. Sugihara, H. I. James, E. J. Troianello and V. T. Bowen (Dept. of Chem., Clark Univ., Worcester, Mass., U.S.A.). *Anal. Chem.*, 1959, **31** (1), 44–49.—Procedures are given for separating and determining ^{90}Sr, ^{137}Cs, ^{144}Ce and ^{147}Pm in 100 to 200 litres of sea water. The carbonates in the sample (adjusted to pH 7 and containing Sr, Cs and Fe^{3+} as carriers) are pptd. with 5% Na_2CO_3 soln. The supernatant liquid, adjusted to pH 2, is the Cs fraction (*A*); the ppt. is dissolved in conc. HCl and $Fe(OH)_3$ is pptd. with conc. aq. NH_3, the filtrate forming the Sr fraction (*B*). The $Fe(OH)_3$ is re-dissolved and is re-pptd; the filtrate is added to *B*, whilst the ppt. (*C*) contains the rare-earth elements. These three fractions receive further treatment. The mixed carbonates of Sr and Ca contained in fraction *B* are pptd. at pH 7 with Na_2CO_3 in the presence of EDTA (tetrasodium salt), dissolved in HNO_3, and $Sr(NO_3)_2$ is then pptd. with 76% HNO_3 after scavenging with $Fe(OH)_3$ - $BaCrO_4$. The Sr is converted into $SrCO_3$ and the activity of ^{90}Sr is determined on ^{90}Y (separated after ≃ 15 days). After co-pptn. of the alkali metals in fraction *A* with dipotassium sodium cobaltinitrite, the ppt. is dissolved in hot 6 *N* HCl, the Cs is pptd. as tungstosilicate, converted into perchlorate and then separated from ^{40}K and ^{87}Rb by an ion-exchange procedure. The rare-earth elements in fraction *C* are re-pptd. on $Fe(OH)_3$; U^{6+}, Pa^{5+} and Th are then removed by ion exchange, and radium isotopes and ^{210}Pb by scavenging with $BaSO_4$. Finally, pure ^{144}Ce is obtained by separation as Ce^{4+} and pure ^{147}Pm by cation exchange (Nd is added to the Pm fraction to reduce self-absorption); β-absorption curves are determined for Cs, Ce and Pm (polyethylene absorbers are used). The low activities (≃ 0·1 to 1 c.p.m.) are measured with a small-flow Geiger counter (Sugihara *et al.*, *Rev. Sci. Instrum.*, 1953, **24**, 511). Chemical yields are 50 to 80%, and the blank correction for decontamination is negligible. W. J. Baker

3250. **Determination of radio-ruthenium in effluent and sea water.** B. A. Loveridge and A. M. Thomas (A.E.R.E., Harwell, Berks., England). A.E.R.E. Report C/R 2828, 1959, 11 pp.—The sample (up to 1 litre) is heated near the b.p. for several minutes with ruthenium carrier and KOH. The soln. is cooled and acidified, the RuO_4 is extracted with carbon tetrachloride (3 × 10 ml). The Ru is then pptd. as the dioxide and oxide by the addition of a little alcohol. The ppt. is β-counted and then dissolved in alkaline periodate soln.; the extinction of this soln. is a measure of the chemical yield of the separation. The recoveries of ^{106}Ru added in various forms to sea water in 15 experiments were in the range 95 to 105%. G. J. Hunter

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See also Abstracts—**2825**, Air pollution (review). Water analysis (review). **2847**, Determination of Cl^- in water. **2854**, Micro-determination of gases in water. Determination of F^- in drinking water. **3276**, Apparatus for determination of O in boiler feed water.

Agricultural analysis

Soil, fertilisers, herbicides, pesticides, animal feeding-stuffs.

3251. **Methods of taking and preparing samples of soil.** F. Dugain. *Fruits d'Outre Mer*, 1958, **13** (9–10), 395–400.—Determinations of organic C, organic N and humus on the soil of an area of 140 sq. metres, required, respectively, 22, 15 and 40 samples for a precision of 5%. Concn. may vary markedly in adjoining small areas, and also at different depths. It is necessary to take large numbers of small samples as rapidly and easily as possible. Sampling tubes found useful for clay and sandy soils are illustrated. At the laboratory, the samples from one area are combined, dried, sieved and homogenised, and divided into aliquots by a special "sample-divider" apparatus. M. D. Anderson

3252. **Determination of nitrogen in water-logged soils.** J. M. Bremner and K. Shaw (Rothamsted Exp. Sta., Herts., England). *J. Agric. Sci.*, 1958, **51**, 22–39.—For the determination of total N in these soils (which may contain NO_3^-, NO_2^-, NH_4^+, etc.), Olsen's method (*Compt. Rend. Lab. Carlsberg*, 1929, **17**, No. 3) is modified. The soil (5 to 20 g containing up to 100 mg of N), together with water (10 ml), 5% (w/v) $KMnO_4$ soln. (10 ml) and 50% (w/v) H_2SO_4 (20 ml), is placed in a Kjeldahl flask, the neck of which is covered with an inverted Erlenmeyer flask, and the contents are shaken. After 5 min., 2 drops of 2-ethylhexanol and 5 g of reduced iron (100 mesh) are added. After the initial reaction has subsided (15 min.) the flask is heated gently for 15 min., then the covering flask is removed and K_2SO_4 (10 g), $CuSO_4$ (1 g), Se (0·1 g) and H_2SO_4 (40 ml) are added and the digestion and distillation are continued in the normal way. For the separate determination of NO_3^-, NO_2^- and NH_4^+, the soil is extracted with *N* KCl or *N* K_2SO_4, and an aliquot, used for NO_3^- and NH_4^+, is treated with sulphamic acid (2%, 1 ml) to avoid interference by NO_2^-, NH_4^+ being determined by the Conway diffusion method. Nitrite is determined separately by Shinn's method (*Ind. Eng. Chem., Anal. Ed.*, 1941, **13**, 33).

A. G. Pollard

3253. **Determination of fixed ammonium in soils.** A. P. S. Dhariwal and F. J. Stevenson (Univ. Illinois, Urbana). *Soil Sci.*, 1958, **86**, 343–349.—The sample of clay (1 g) or finely ground soil (2 g) is autoclaved with 50 ml of *N* KOH for 8 hr. at 120° to remove exchangeable NH_4^+ and org. matter. For highly calcareous soils the sample is suspended in *N* KCl, and HCl is added dropwise until carbonates are decomposed, the residual soil being recovered centrifugally before the KOH treatment. The autoclaved material is centrifuged and washed with 0·5 *N* KCl (3 × 20 ml); 5 *N* HF (15 ml) is added and the mixture stirred occasionally for 12 to 16 hr. It is then transferred to a micro-Kjeldahl distillation unit containing sufficient NaOH to make the mixture approx. *N* to NaOH to avoid contamination of distillate with HF, NH_3 being then distilled in the normal way. The ammonium-fixing capacity of the soil is determined similarly after the sample had been treated with NH_4Cl (4 milli-equiv. in 1 ml) and dried at 30°.

A. G. Pollard

3254. **Determination of water-soluble sulphate in soils.** J. R. Freney (C.S.I.R.O., Australia). *Soil Sci.*, 1958, **86**, 241–244.—The soil sample (20 g) is shaken with sulphur-free water (100 ml) in a centrifuge bottle. After 30 min. the suspension is centrifuged (2000 r.p.m.) and the supernatant liquid is filtered through a Seitz filter (Grade D 9). The filtrate (2 ml) is acidified with 0·01 *N* HCl (to methyl orange) and boiled. Aq. $BaCl_2$ soln. (5%) is added dropwise to the boiling liquid and the vessel is kept warm for 1 hr. The supernatant liquid is removed by a filter-stick (sintered glass, porosity 4) the vessel and ppt. being washed several times with 1% $BaCl_2$ in 0·05 *N* HCl. The final determination is made by the method of Johnson and Nishita (*cf. Brit. Abstr. C*, 1952, 356).

A. G. Pollard

3255. **Determination of ferrous iron in paddy soils.** K. Kumada and J. Asami (Fac. Agric., Univ. Tokio). *Soil and Plant Food, Tokyo*, 1958, **3**, 187–193.—To correct for oxidation of Fe^{2+} during the sampling of the wet soil, a portion corresponding to 1 to 4 g of dry soil is placed in a small test-tube and covered with 4 ml of water. The tube is then sealed and incubated at 40° for 10 days. It is then broken open and the soil transferred to a 100-ml flask containing 40 ml of *N* Na acetate buffer (pH 2·8), a portion of the buffer being used for the transference. After shaking the flask for 2 hr., the contents are diluted to the mark and filtered through a dry paper. Ferrous iron is determined by the 2:2′-dipyridyl method.

A. G. Pollard

3256. **Apparent loss of organic nitrogen in fertilisers containing urea and natural organics.** C. E. Waters and R. G. Ziegler (Nitrogen Div., Allied Chemical Corp., Hopewell, Va., U.S.A.). *J. Agric. Food Chem.*, 1958, **6** (11), 833–838.—Analyses by standard methods of fertilisers containing urea (**I**) and organic material (leather tankage, activated sewage sludge, etc.) often give lower contents of organic N than those calculated. Analyses of organic materials and other fertiliser ingredients stored with or without **I** showed that breakdown of **I** was generally small, though an original **I** content of 2·2% fell to 1·8% after 5 weeks at 50°. Values for ammoniacal N were often high in the presence of **I**, because of hydrolysis of part of the **I** during distillation from a slurry of MgO; owing to the methods of calculation employed, this gives a low apparent organic N. The error can be minimised by not prolonging the distillation. The use of a low-temp. procedure for the determination of NH_3 is suggested.

M. D. Anderson

3257. **Determination of indol-3-ylacetic acid by the Salkowski reaction.** A. M. Mayer (Botany Dept., Hebrew Univ., Jerusalem). *Nature*, 1958, **182**, 1670–1671.—The Tang-Bonner reaction (*Arch. Biochem.*, 1947, **13**, 11) has been modified by dissolving the $FeCl_3$ in trichloroacetic acid. The absorption maximum of the interaction product between Fe^{3+} and indol-3-ylacetic acid is at 545 mμ and the relationship between concn. and extinction is linear up to 80 μg of acid per 5 ml of reaction mixture. The determination of the Fe^{2+} produced demonstrated the quant. reduction of Fe^{3+} by the indol-3-ylacetic acid.

S. Baar

3258. **Identification of translocated regulating chemicals through plant response and chromatography.** B. C. Smale and E. J. Daly (Crops Res. Div., Agric. Res. Service, U.S. Dept. of Agric., Beltsville, Md.). *J. Agric. Food Chem.*, 1958, **6** (10), 751–752.—The method described for locating plant-regulating substances, after absorption and translocation in a plant, involves extraction of the plant material, chromatography of the extract on paper, elution of separated substances with ether and lanolin, removal of ether, and application of the lanolin to a particular spot on young bean plants at a well-defined stage of growth. The different regulating substances cause characteristic responses. The method is suitable both for compounds that affect cell elongation (2:4-dichlorophenoxyacetic, indol-3-ylacetic and gibberellic acids), and those that cause modification of leaf development (α-methoxyphenylacetic and 2:3:5-tri-iodobenzoic acids).

M. D. Anderson

3259. **Polyethylene-alumina column for purification of tissue extracts before analysis.** W. M. Hoskins, W. R. Erwin, R. Miskus, W. W. Thornburg and L. N. Werum (Dept. Entomol. Parasitol.,

Univ. California, Berkeley). *J. Agric. Food Chem.*, 1958, **6** (12), 914–916.—Columns containing granules of alumina coated with wax have been used for purification of plant and animal extracts before determination of residual pesticides, but have the disadvantage that the wax is slightly sol. in the methyl cyanide used as eluent. Granules of alumina were coated with polyethylene (dissolved in boiling toluene), and columns of these granules gave satisfactory results with several plant products and insecticides. Retention of extractives on the column ranged from 30% for extracts of tomato purée to 95 to 99% for lucerne extracts; the recovery of insecticides was 81 to 100%.

M. D. ANDERSON

3260. **Semi-micro quantitative analysis of copper 8-quinolinate. Demonstration of uneven deposition of fungicide on cellulosic materials as a major variable in deterioration evaluation.** A. D. Baskin, J. Bubernak, S. P. Reynolds, J. B. Oakes and H. D. Lyster (U.S. Army Quartermaster Res. and Engng Center, Natick, Mass.). *Amer. Dyest. Rep.*, 1958, **47** (18), 603–608.—Anomalies have been observed between the measured concn. of fungicide and loss in breaking strength after mildew-resistance tests on cotton textiles. Semi-micro quant. modifications of the methods described by (*i*) Rose *et al.* (*cf. Anal. Abstr.*, 1957, **4**, 969) and (*ii*) Woelfel (*Anal. Chem.*, 1948, **20**, 723) for the analysis of Cu 8-quinolinate have been devised, in order to study the evenness of deposition of the fungicide. (*i*) A 3 to 4-mg sample is extracted successively at 70° to 80° with 3 ml of 10% H_2SO_4 plus 3 drops of $CHCl_3$, 2 ml of 10% H_2SO_4 plus 2 drops of $CHCl_3$, and 2 ml of 10% H_2SO_4 plus 3 ml of H_2O. The extracts are heated to remove $CHCl_3$ and then bulked. The bulk extract is adjusted to pH 6·5 potentiometrically with conc. aq. NH_3 and then extracted successively with 4-, 3- and 3-ml portions of $CHCl_3$. The extracts are bulked over anhyd. Na_2SO_4, filtered and made up to 10 ml. The extinction of the extract is then measured in a spectrophotometer at 410 mμ in a 1-cm cell, against $CHCl_3$ as the blank, and results are referred to a standard curve. (*ii*) The material (3 to 4 mg) is digested in a micro Kjeldahl flask with 0·4 ml of H_2SO_4 (1:1), and a few drops of H_2O_2 (30%) to hasten oxidation. The digest is diluted to 10 ml with double-distilled water, 1·2 ml of 12 *N* HNO_3 is added and the mixture is neutralised with 15 *N* aq. NH_3, then made just acid to litmus with 1 drop of HNO_3. After the addition of 2·5 ml of acetate buffer and 0·25 ml of Cuprethol reagent [diethanolamine bis(2-hydroxyethyl)dithiocarbamate], the soln. is diluted to 10 ml and the extinction measured at 434 mμ in a spectrophotometer, against an extract of untreated material. The amount of fungicide present is obtained from a standard curve. Results for various treated cotton threads are detailed and discussed. The semi-micro methods demonstrate that the deposition of fungicide is uneven. It is possible that this unevenness may be an important variable in itself, apart from the method of analysis and size of sample, and may contribute to the poor agreement observed between determinations within and between laboratories.

S. M. MARSH

3261. **Determination of N-*m*-tolylphthalamic acid residues in food crops.** J. R. Lane, D. K. Gullstrom and J. E. Newell (Naugatuck Chemical, Div. U.S. Rubber Co., Naugatuck, Conn.). *J. Agric. Food Chem.*, 1958, **6** (10), 753–754.—N-*m*-Tolylphthalamic acid (Duraset) residues are determined at the 0·1-p.p.m. level by distillation of an homogenised sample from zinc and NaOH to liberate *m*-toluidine, which is collected in aq. acid, diazotised with HNO_2, and coupled with N-naphthylethylenediamine to give a magenta colour, which is measured at 560 mμ in 10-cm cells. Recoveries of 0·1 p.p.m. of Duraset added to tomatoes, cherries and strawberries averaged, respectively, 89 ± 9%, 79 ± 12%, and 85 ± 13%. Background interference values for untreated samples averaged 0·05 to 0·08 p.p.m. of Duraset.

M. D. ANDERSON

3262. **Spectrophotometric determination of pentachloronitrobenzene on food and forage crops.** H. J. Ackermann, H. A. Baltrush, H. H. Berges, D. O. Brookover and B. B. Brown (Ind. Chem. Div., Olin Mathieson Chem. Corp., New Haven, Conn.). *J. Agric. Food Chem.*, 1958, **6** (10), 747–750.—Residues of the soil fungicide pentachloronitrobenzene (Terraclor) are determined by a modification of Canback and Zajaczkowska's photometric method for organic nitro compounds (alkaline hydrolysis to KNO_2, which is then used to diazotise procaine hydrochloride; this is coupled with 1-naphthylamine to give a magenta-coloured soln., the absorption of which is measured at 525 mμ). Quantities in the range 10 to 15 μg, or as low as 0·02 p.p.m., can be detected. Slightly different procedures are described for green vegetables, forage crops, and oil-seeds. Residues of Terraclor in food and forage crops were very low when dosages and methods of application had been those recommended.

M. D. ANDERSON

3263. **Separation and identification of chlorinated organic pesticides by paper chromatography. XI. A study of 114 pesticide chemicals: technical grades produced in 1957 and reference standards.** L. C. Mitchell (Div. Food, Food and Drug Admin., Dept. of Health, Educ. and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (4), 781–816.—R_F values are given for 213 specimens of 114 different pesticide chemicals. Two solvent systems were employed—(*i*) stationary, soya-bean oil in diethyl ether; mobile, aq. 2-methoxyethanol; (*ii*) stationary, 2-phenoxyethanol in diethyl ether; mobile, 2:2:4-trimethylpentane. The chromogenic agent was $AgNO_3$ with 2-phenoxyethanol in aq. acetone, followed by exposure to u.v. light. Values are tabulated.

A. A. ELDRIDGE

3264. **Colorimetric determination of heptachlor in soils and some crops.** E. P. Lichtenstein and K. R. Schulz (Dept. of Entomol., Univ. Wisconsin, Madison, U.S.A.). *J. Agric. Food Chem.*, 1958, **6** (11), 848–849.—In the extraction of heptachlor from soils and crops for determination by the method of Polen and Silverman (*cf. Brit. Abstr. C*, 1952, 403), Soxhlet extraction with pentane, followed by passage through a 10-g Florex column), gave low and erratic recoveries. Extraction by shaking with a mixture of pentane and acetone (4 : 1), followed by treatment of the soln. with activated carbon, gave consistent recoveries of ≃ 90%, and less pentane was necessary.

M. D. ANDERSON

3265. **Determination of pyrethrins and other insecticidal compounds by infra-red spectrometry.** N. G. McTaggart, E. Thornton and A. D. Harford (Brit. Petroleum Co. Ltd., Res. Sta., Sunbury-on-Thames, Mddx., England). *Pyrethrum Post*, 1958,

4 (4), 12–15.—Details are given of modified procedures in the application of the differential i.r. technique, with a double-beam spectrometer, to the determination of pyrethrins and other materials in preparations of insecticides. Synthetic blends of insecticides were prepared and analysed. Results obtained for blends and extracts show that the method is adequate for checking anomalous biological results, for blending control, and for rapid quality checks of the active ingredients used in formulation. S.C.I. Abstr.

3266. **A new colour reaction for dieldrin and endrin.** E. J. Skerrett and E. A. Baker (Long Ashton Res. Sta., Univ. Bristol, England). *Chem. & Ind.*, 1959, (17), 539.—*Procedure*—Benzene soln. of dieldrin or endrin are isomerised by heating with BF_3 - diethyl ether complex to the 6-oxo derivatives. A red colour is produced by treating the 2:4-dinitrophenylhydrazone of this ketone with tetraethylammonium hydroxide. Under controlled conditions 10 μg is detectable, and 1 mg of DDT or BHC does not interfere. E. J. H. Birch

3267. **Determination of the plant-protection product metaSystox-i [demeton-S-methyl]. I. Determination in technical products and formulations.** W. Pilz (Farbenfabrik. Bayer A.-G., Leverkusen, Germany). *Z. anal. Chem.*, 1958, **164** (2), 241–246.—The insecticide is determined in technical products by saponification in ethanol with plumbite soln.; this fixes the unstable mercaptan first formed as a complex lead salt, extractable with $CHCl_3$. Then (*i*) the mercaptan is liberated with H_2SO_4 and titrated iodimetrically or (*ii*) the Pb is extracted from the complex with HCl, boiled until free from mercaptan and determined by adding aq. NH_3 - NH_4Cl buffer, Eriochrome black T and excess of EDTA, and back-titrating with standard zinc soln. Maximum deviations are, respectively, $\pm$0·6% and $\pm$0·3%. P. D. Parr-Richard

3268. **Colorimetric method for estimation of guthion residues in cottonseeds and cottonseed oil.** P. A. Giang and M. S. Schechter (Entomology Res. Div., Agric. Res. Service, U.S. Dept. of Agric., Beltsville, Md.). *J. Agric. Food Chem.*, 1958, **6** (11), 845–847.—The insecticide guthion is determined in cottonseeds and cottonseed oil by extracting with $CHCl_3$, evaporating the solvent, dissolving the residue in pentane, separating the guthion on a column of activated alumina, eluting with methyl cyanide, evaporating the solvent, and hydrolysing the guthion with HCl; the formaldehyde formed is distilled off and determined colorimetrically with chromotropic acid at 570 mμ.

M. D. Anderson

3269. **Colorimetric micro-determination of the acaricide 2:4:5:4′-tetrachlorodiphenyl sulphone [Tedion].** O. H. Fullmer and C. C. Cassil (Niagara Chem. Div., Food Machinery and Chemical Corp., Richmond, Calif.). *J. Agric. Food Chem.*, 1958, **6** (12), 906–908.—Residues of Tedion, in the range 5 to 50 μg, are determined in plant material by extracting with $CHCl_3$ containing 1 g of lanolin per 100 ml (for fruit), or benzene (for orange juice), evaporating to dryness, chilling, nitrating, neutralising with KOH, extracting with $CHCl_3$, carefully removing all solvent, dissolving in pyridine containing 4% of water, and adding KOH, when a red colour is produced, the extinction of which is measured at 520 mμ and referred to a standard curve. DDT interferes, and DDD to a lesser extent; other insecticides do not.

M. D. Anderson

See also Abstracts—**2825**, Reviews of analysis of fertilisers and pesticides. **2869**, Separation of ^{137}Cs in soil. **2924**, Determination of P. **3034**, Determination of biuret in fertilisers. **3096**, Determination of parathion in blood. **3219**, Determination of Sevin in wine.

5.—GENERAL TECHNIQUE AND APPARATUS

General

3270. **Device for opening small sealed objects under vacuum.** G. P. Schacher (General Engng Lab., General Electric Co., Schenectady, N.Y.). *Anal. Chem.*, 1959, **31** (1), 161.—The object (*e.g.*, a sample of gas in an hermetically sealed container) is held against a support inside an evacuable tube by means of a steel point which operates through a stainless-steel bellows. It is punctured by turning a threaded rod which applies pressure to the steel point. This device has been used in conjunction with a mass spectrometer for determining the gaseous composition of samples $<$ 1 cu. mm in vol. at pressures of $<$ 0·5 atm. A. R. Rogers

3271. **Improvements in or relating to burettes.** M. C. Sanz. Brit. Pat. 811,838, date appl. 12.11.57. Switzerland, date appl. 20.11.56.—A burette for delivering and indicating variable small amounts of liquid delivered, comprises a reservoir into which a rod extends and is adapted to displace liquid from the reservoir. A micrometer assembly moves the rod a predetermined axial distance for each revolution of the micrometer screw. A wire wound on to an element, which rotates with the screw, drives an indicator dial to show the amount of liquid displaced from the reservoir by the rod.

J. M. Jacobs

3272. **Molecular sieves as gas samplers.** B. L. Tonge and D. G. Timms (Gen. Electric Co. Ltd., Wembley, England). *Chem. & Ind.*, 1959, (5), 155–156.—Linde molecular sieves No. 5A and 13X, 36 to 52 B.S. mesh, activated by heating *in vacuo* to 300° and then cooled in liquid air for $\simeq$ 1 hr. (residual pressure $< 10^{-5}$ mm), can be used to remove gas from a system under low pressure. The gas can be collected into a small vessel containing the molecular sieve, which can then be removed and transferred to a gas analyser or mass spectrometer. Application to gas analysis in a low-pressure system by gas chromatography is described briefly. W. J. Baker

3273. **Gas-blending apparatus.** D. N. Hanson and A. Maimoni (Univ. of Calif. Radiation Lab., Berkeley, U.S.A.). *Anal. Chem.*, 1959, **31** (1), 158–159.—The apparatus described can be used to prepare synthetic gas mixtures of any composition with an accuracy of about 0·01%.

K. A. Proctor

3274. **Diffusion by "sweeping". Application to the separation of the constituents of a gaseous mixture.** J. Bernard (Lab. de Chimie Minérale, Fac. des Sci., Paris). *J. Chim. Phys.*, 1958, **55** (10), 844–859.—A review is presented of those

methods of, for example, isotope concentration, in which the gaseous mixture and a carrier gas are passed on either side of a barrier having holes in it greater than the mean free path of the molecules in the gas (double diffusion technique). (14 references.)
T. R. ANDREW

3275. **Improvements in or relating to apparatus for detecting the presence of explosive or combustible gases and vapours.** C. McLuckie. Brit. Pat. 811,796; date appl. 1.5.56.—The apparatus described in Brit. Pat. 319,231 (comprising two chambers in one of which a sample of the atmosphere to be tested is ignited electrically, so that any combustible gases are burnt, the difference in pressure after the chambers have again attained the same temp. being indicated on a manometer) is provided with means for effecting saturation with water of the atmosphere in the two chambers.
J. M. JACOBS

3276. **Improvements in or relating to methods and apparatus for detecting and/or measuring the concentration of oxygen in aqueous liquids such as boiler feed water.** Engelhard Industries, Ltd. [Inventors: A. G. Dowson and I. J. Buckland]. Brit. Pat. 806,918, date appl. 25.8.55.—The liquid, the O content of which is to be measured, is caused to flow from a chamber and down a pipe where it is brought into contact with an inert carrier gas (H) so that some of the O in the liquid is transferred to the gas. The liquid and the gas are then separated and the gas is introduced into a galvanic cell (having a cathode of silver wire, an anode of finely divided cadmium, and an electrolyte of 5% aq. KOH soln.) where the O is absorbed at the cathode to produce an electric current which is a function of the O concn. in the ambient gas in contact with the cell.
J. M. JACOBS

3277. **Method of indicating excessive quantities of water in liquids of low water solubilities.** Ansul Chem. Co. Brit. Pat. 810,947; date appl. 14.3.57. U.S.A., date appl. 26.11.56.—A method for testing whether the content of water in halogenated refrigerants, etc., is just above or just below the range of 1·2 to 2·4% relative saturation comprises the immersion in the test liquid of an indicating material consisting of a mat of inert non-cellulosic fibre upon which predetermined amounts of pure anhyd. cobaltous bromide are deposited. The colour of the indicator is purple red - purple (Munsell colour [10·0 P] 7/8) just above, and yellow - green (Munsell colour [2·5 G] 7/8) just below, the range.
J. M. JACOBS

3278. **Determination of the viscosity of liquids in c.g.s. units.** British Standards Institution (2 Park Street, London). B.S. 188:1957. Amendment No. 1, published 18.2.59, 5 pp.—In addition to minor amendments, the table of calculated B-values has been revised and the three drawings of viscometers have been changed. (*Cf. Anal. Abstr.*, 1957, **4**, 1407.)

3279. **[Symposium on] the accurate measurement of the viscosity of liquids.** R. Roscoe, M. T. Browne *et al.* *J. Inst. Petrol.*, 1958, **44**, 394–400.—Seven papers (summarised) on the accuracy and limitations of different viscometric procedures relate to (*i*), the oscillating-vessel method, in which increased accuracy is attainable with a cylindrical vessel and an improvement in measuring the logarithmic decrement; (*ii*), the electronic counting technique, in which viscosity is calculated from the measured damping of a torsionally vibrating metal rod immersed in the sample; small changes (*e.g.*, $\simeq 1\%$) in viscosity can be determined to within $\simeq 5\%$; (*iii*), the effect on the coeff. of viscosity of altering the rate of shear; a jet viscometer can provide an assessment, in $\simeq 500$ hr., of the degree of shear breakdown in a polymer-thickened hydraulic oil; (*iv*), measuring the viscosity of polymers at very low concn. with a U-tube capillary apparatus; (*v*), measuring the kinematic viscosity of heavy oils at high and low temp.; (*vi*), standard methods of measuring viscosity in c.g.s. units, with reference to the instruments, procedures and calculations specified in B.S. 188:57 and IP 71/58; and (*vii*), the difficulty of using the quadratic equation for calculating the coeff. of viscosity from flow times.
W. J. BAKER

3280. **Improvement of the Weygand and Werner apparatus for quantitative hydrogenation.** H. Wetzel (Lab. VEB Chem. Fabrik Miltitz, Leipzig, Germany). *Z. anal. Chem.*, 1958, **164** (4), 407–408.—The accuracy of results obtained with the original apparatus (*J. prakt. Chem.*, 1937, **149**, 330) has been increased by improved arrangements for stirring, introducing the sample and measuring the gas in the burette.
W. J. BAKER

Chromatography, ion exchange, electrophoresis

3281. **Constant-volume fraction collector for column chromatography.** W. J. Wechter, J. E. McCarty and B. E. Fisher (Dept. of Chem., Univ. of Calif., Los Angeles). *Anal. Chem.*, 1959, **31** (1), 159–160.—Automatic collection of fractions of from 7 to 500 ml can be made of almost any solvent used in chromatography. When the receiver fills to the appropriate vol., a silvered glass float interrupts a light beam between a lamp and a photocell. Relays are operated which empty and drain the receiver through a glass-enclosed ball-and-socket valve, and which then rotate the table assembly.
D. W. Moss

3282. **Applications of chemisorption in chromatographic separations of organic isomers and homologues.** J. L. Forstner and L. B. Rogers (M.I.T., Cambridge, Mass., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 365–370.—The advantages of using chemisorption instead of physical sorption for increased selectivity in chromatographic separations have been studied, with 2- and 4-nitroso-1-naphthols. Low-capacity salts and impregnated high-capacity oxides are effective as chromatographic sorbents.
K. A. PROCTOR

3283. **Paper chromatography in the separation of ions. IV.** A. K. Majumdar and A. K. Mukherjee (Jadavpur Univ., Calcutta, India). *Anal. Chim. Acta*, 1958, **19** (5), 480–482.—Five solvent mixtures for the development of inorganic chromatograms are studied. The mixture advocated is *tert.*-butyl alcohol - H_2O - HCl (sp. gr. 1·19) (7:2:1). It is possible to separate Li^+ from other alkali metals; Mg^{2+} from the alkaline earths; Zn^{2+} and Mn^{2+} from Co^{2+} or Ni^{2+}; Fe^{3+}, Be^{2+}, Ti^{4+}, UO_2^{2+} (or Cr^{3+} or Al^{3+}) and Ce^{3+} (or Th^{4+}) from ZrO^{2+} (or Nb^{5+} or Ta^{5+}); VO_3^-, AsO_4^{3-} and WO_4^{2-} and also PO_4^{3-}, CrO_4^{2-} and MoO_4^{2-}.
T. R. ANDREW

3284. **Electrostatic discharge methods of zone location for paper chromatographs. III. Further developments.** G. G. Blake (Dept. of Chem., Sydney Univ., Australia). *Anal. Chim. Acta*, 1958, **19** (5), 477–479.—A modification of earlier apparatus (*cf. Anal. Abstr.*, 1958, **5**, 3198) permits the use of any type of triode valve by application of a negative grid bias. An automatic zone locating and marking device is described. T. R. ANDREW

3285. **Simple device for the simultaneous development of several two-dimensional chromatograms.** T. Borkowski and S. Trojnar (Sch. of Med., Lublin, Poland). *J. Chromatography*, 1958, **1** (6), 552–553.—The device, a wooden cross-shaped stand with pointed parallel glass rods attached to the ends of the arms, is especially useful when it is necessary to maintain identical conditions (*e.g.*, size of chamber, saturation, temp. and duration of development) throughout an experiment involving several chromatograms. G. P. COOK

3286. **Separation by gas - liquid chromatography.** G. W. A. Rijnders (Konikl./Shell Lab., Amsterdam, Netherlands). *Chem. Weekbl.*, 1958, **54** (48), 669–674.—The basic principles of gas - liquid chromatography are discussed. The influence of the polarity of the liquid stationary phase and of the physical properties of the solid support is discussed in relation to the mixture to be separated. The separation of mixtures of hydrocarbons is described. G. BURGER

3287. **Inexpensive gas-chromatography apparatus.** P. Geldenhuis, W. Nel and V. Pretorius (Dept. of Phys. Chem., Univ., Pretoria, S. Africa). *S. Afr. Ind. Chem.*, 1958, **12** (10), 196–197.—The apparatus described can be used for the analysis of most low-boiling organic liquids. With 10-μl samples of benzene the 95% confidence limits of the measured peak heights were $\pm$ 1·02% (16 tests). The modified flame detector used has two hydrogen jets, each with its own thermocouple. One jet is connected directly to the column so that any change in the column eluate results in an e.m.f. between the two thermocouples which is measured on a sensitive millivoltmeter. K. A. PROCTOR

3288. **Circular gas chromatograph.** R. S. Porter and J. F. Johnson (California Res. Corp., Richmond, U.S.A.). *Nature*, 1959, **183**, 391–392.—By using the circular apparatus described it is possible to employ, as stationary phases, liquids which do not have a low volatility relative to the compounds being separated. In this way a wider range of materials can be utilised. As the sample can be cycled a number of times this arrangement offers the possibility of operating the equivalent of a long column at a low and more efficient ratio of inlet and outlet pressures. In a circular 10-ft. column with 2:4-dimethylsulpholane as the stationary phase, the efficiency increased linearly with the number of cycles up to a column length equiv. to 150 ft. The equipment is also useful on a preparatory scale and can be made automatic. K. A. PROCTOR

3289. **Automatic control of gas chromatography for volumetric measurement.** J. Janák and K. Tesařík (Lab. for Gas Analysis, Czech. Acad. of Sci., Brno). *Z. anal. Chem.*, 1958, **164** (1), 62–69.—This is based on the direct reading of the vol. of the components at constant pressure. A sensitive contact manometer is used to detect changes in pressure in the nitrometer. Diagrams of the nitrometer and contact manometer are shown. The practical application of the apparatus is demonstrated by the analysis of a propane - butane mixture, and the average error for a 2-ml sample is 0·4% and for 5 ml $<$ 0·25%, by vol. B. B. BAUMINGER

3290. **Selection of stationary phase for qualitative gas-chromatographic analysis.** G. Raupp (Bodensee-werk Perkin-Elmer & Co., Überlingen/See). *Z. anal. Chem.*, 1958, **164** (1), 135–146.—The retention vol. (*n*-pentane = 1) of 120 organic compounds with boiling-points of from 30° to 220° have been determined with 6 different stationary phases. The degree of separation depends mainly on the chemical nature and polarity of the stationary phase. Some chromatograms show that (*i*) fluorene - picric acid is suitable for the analysis of aromatic compounds, (*ii*) di-(2-ethylhexyl) sebacate, di-*n*-decyl phthalate, silicone oil and tetraethylene glycol dimethyl ether are satisfactory for paraffins, and (*iii*) polyethylene glycol for the analysis of alcohols. B. B. BAUMINGER

3291. **3:3′-Oxydipropionitrile [di-(2-cyanoethyl) ether] and hexaethylene glycol dimethyl ether as stationary phases for gas chromatography.** H. Kelker (Farbwerke Hoechst A.-G., Frankfurt/Main, Germany). *Angew. Chem.*, 1959, **71** (6), 218–220.—3:3′-Oxydipropionitrile and hexaethylene glycol dimethyl ether are valuable as stationary phases; they have good selective powers for a large number of aliphatic compounds and are also suitable for the analysis of aq. soln. The relative retention volumes at 70° for $\simeq$80 compounds are listed. G. P. COOK

3292. **The chromatography of gases and vapours. VI. Use of stearates of bivalent manganese, cobalt, nickel, copper and zinc as column liquid in gas chromatography.** D. W. Barber, C. S. G. Phillips, G. F. Tusa and A. Verdin (Inorg. Chem. Lab., Oxford). *J. Chem. Soc.*, 1959, (1), 18–24.—The stearates of Mn, Co, Ni, Cu and Zn are liquid at 104°, 92°, 146°, 101° and 122°, respectively, and for chromatography are mixed (19 parts) with Celite (100 parts). The columns are contained in glass tubes (4 mm $\times$ 100 cm) and N - H (1:3) is used as a carrier gas. Relative retention times on these stearates and Apiezon L grease are measured at 156°, with mesitylene as internal standard. Plots of relative retention rates on Apiezon L against those on the metal stearates give straight lines for each homologous series. The slopes of these lines are called the retardation factors for the series, and when plotted against the atomic number of the stearate show a max. at Ni, followed by a sharp fall to Cu for alcohols and ketones; it is pointed out that the pattern is reminiscent of that for the complex stabilities for substitution at the 5th and 6th position on the metal. Amines have a much greater retardation factor which reaches a max. at Co and falls to Ni, and for secondary and tertiary amines on Mn, Co and Zn stearates the factor is proportional to the basic dissociation constant. Thermodynamic data are calculated from the results of experiments at 110·6° and 156°. E. J. H. BIRCH

3293. **Gas - liquid chromatography. Determination of column efficiency.** H. W. Johnson, jun., and F. H. Stross (Shell Devel. Co., Emeryville, Calif., U.S.A.). *Anal. Chem.*, 1959, **31** (3),

357–365.—In gas-liquid chromatography it is essential to separate the peak width into column and non-column effects as errors may arise when the entire peak width is attributed to the column alone. The precision of peak widths is compared with that of partition coeff., and a method is described for estimating column peak widths. K. A. PROCTOR

3294. **A capillary tube crusher for use in gas chromatography.** J. Joklík and V. Bažant (Dept. Org. Technol., Chem. Inst. Acad. Sci., Prague). *Chem. Listy*, 1959, **53** (3), 277–278.—The apparatus consists of a brass cylinder which can be heated to a desired temp.; the capillary tube containing the sample is placed in a part of the cylinder separated by two taps and after 5 min. is crushed by means of a lever. J. ZÝKA

3295. **New procedure for the collection of fractions in gas chromatography.** J. H. Jones and C. D. Ritchie (Div. Cosmetics, Food and Drug Admin., Dept. of Health, Educ. and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (4), 753–756.—The vapour emerging from the column is mixed with the vapour of a suitable solvent, and the condensate fractions, which are collected as in liquid chromatography, are analysed qual. and quant., *e.g.*, by u.v. spectrophotometry. Curves for extinction *vs.* fraction number are given for mixtures of aromatic amines and nitro derivatives. The sensitivity of detection is comparable with that reached by thermal-conductivity methods. A. A. ELDRIDGE

3296. **A detector for gas-liquid chromatography.** R. A. W. Johnstone and A. G. Douglas (Univ., Exeter, England). *Chem. & Ind.*, 1959, (5), 154.—The arrangement described enables polycyclic aromatic compounds, separated by gas chromatography, to be detected by their u.v. absorption. The detector is a silica gas-cell (10 cm long) through which light of $\simeq$250 mμ is passed, the absorption of radiation causing a drop in the photomultiplier output. The temp. of the cell must always be higher than that of the chromatographic column. Limits of detection for naphthalene and diphenyl are 8·8 and 2·1 moles per ml $\times$ 10^{10}, the corresponding detector sensitivities being 385 and 785 ml $\times$ mV per mg. W. J. BAKER

3297. **Detector for use with capillary-tube columns in gas chromatography.** J. E. Lovelock (Nat. Inst. for Med. Res., Mill Hill, London). *Nature*, 1958, **182**, 1663–1664.—To meet the stricter requirements of metal capillary-tube columns (*e.g.*, smallness of load and retention-vol.), the sensitive detector described previously (*cf. Anal. Abstr.*, 1958, **5**, 3208) has been modified to ensure a sensing vol. of $\simeq$ 1 μl. This vol. is obtained by making the anode small in comparison with the ionisation chamber (1 cm $\times$ 1 cm) so that the primary electrons (used to excite the A atoms) are accelerated only in the immediate vicinity of the anode, which is enclosed in a small sensing chamber (1 mm $\times$ 1 mm) open to the main chamber. A scavenging flow of A, dry air or N is passed through the main chamber to confine the response to the sensing chamber. An efficiency of 3 $\times$ 10^4 theoretical plates was obtained in the separation of 0·3 μg of a mixture of hydrocarbons on a column (125 ft. $\times$ 0·01 in.) with a thin layer of squalene as stationary phase. The detector is relatively insensitive to small changes in temp. and pressure, but is slightly sensitive to rate of flow. The response is linear with concn. of vapour provided that a high resistance (1 to 5 $\times$ 10^9 ohms) is included in the source of the applied voltage. W. J. BAKER

3298. **A recording nitrometer detector for gas chromatography.** P. G. Sevenster (Fuel Res. Board, S. Africa). *S. Afr. Ind. Chem.*, 1958, **12** (4), 75–76.—The device described is easily constructed from laboratory equipment and is completely automatic in operation. It can be readily adapted for manual operation and is said to be more sensitive than similar devices. K. A. PROCTOR

3299. **A flame-ionisation detector for gas chromatography.** A. E. Thompson (Dyson Perrins Lab., Oxford). *J. Chromatography*. 1959, **2** (2), 148–154.—The construction of a flame-ionisation detector (*cf. Anal. Abstr.*, 1958, **5**, 4364) and two suitable amplifiers circuits are described. The amplifiers have a very high input and a very low output resistance and are intended for use with the low-resistance recorders normally employed for gas chromatography with thermal-conductivity detectors. A typical amplifier had a signal to noise ratio of 200:1 and a sensitivity for benzene of 1·1 $\times$ 10^6 ml·mV/mg. W. T. CARTER

3300. **Effect of the carrier gas on the sensitivity of a thermal-conductivity detector in gas chromatography.** N. H. Ray (I.C.I. Ltd., Plastics Div., Winnington, Northwich, Ches., England). *Nature*, 1958, **182**, 1663.—It is shown that, notwithstanding the general belief that H and He give the highest sensitivity with a thermal detector (*cf. Anal. Abstr.*, 1956, **3**, 2582), the sensitivity of the detector is inversely proportional to the square of the thermal conductivity of the carrier gas and directly proportional to the difference between the thermal conductivities of gas and organic vapour. Thus, for the same size of sample with He and A as carriers, the sensitivity of A in comparison with that of He is as 2:2:1, respectively, for methane, ethane and propane in a mixture of these. Because the carrier gas with the lowest thermal conductivity has the highest sensitivity, CO_2 and N_2O (among the inorganic gases) should give the best results with thermal-conductivity detection. W. J. BAKER

3301. **Quantitative evaluation of fractograms in gas chromatography. Thermal-conductivity method.** G. Schomburg (Max-Planck-Inst. f. Kohlenforsch., Mülheim-Ruhr, Germany). *Z. anal. Chem.*, 1958, **164** (1), 147–158.—Determinations of area and height of peaks for various aliphatic and aromatic compounds are described in detail, and results are given in tabular form. B. B. BAUMINGER

3302. **Identification and quantitative estimation of gas-chromatography fractions by vapour-phase infra-red spectroscopy.** D. M. W. Anderson and J. L. Duncan (Dept. of Chem., Univ., Edinburgh). *Chem. & Ind.*, 1958, (50), 1662.—An accuracy within $\pm$ 2% can be attained in the i.r. spectrometric determination of microgram amounts of gas-chromatography fractions (b.p. $\gtrsim$ 175°) by the use of cylindrical gas-cells (internal vol. 52 to 110 ml). These cells can be positioned in the sample and reference beams of the Hilger H800 spectrometer. The lower limit of identification and quant. determination is $\simeq$ 10^{-9} mole. Heated

cells can be used for liquids of b.p. $> 175°$. The full procedure is described elsewhere (*cf.* Anderson, *Analyst*, 1959, **84**, 50). W. J. BAKER

3303. **High-temperature gas chromatography of aromatic hydrocarbons.** R. A. Baxter and R. T. Keen (North American Aviation, Inc., P.O. Box 309, Canoga Park, Calif.). *Anal. Chem.*, 1959, **31** (3), 475–476.—Modifications to a commercial gas chromatograph for operation up to 445° are described. Polyphenyls have proved to be useful stationary phases for high-temp. working.
K. A. PROCTOR

3304. **Substitution of Podbielniak distillation by a combination of gas chromatography and gravimetry.** U. Schwenk and E. Weber (Farbwerke Hoechst A.-G., Frankfurt/Main, Germany). *Z. anal. Chem.*, 1958, **164** (1), 159–164.—A method is given for the analysis of gas samples by gas chromatography, thus replacing distillation and analysis in an Orsat apparatus. The time for the analysis of a technical gas mixture is reduced from 6 to < 2 hr. The C_4 fractions are separated in the usual manner, and by reversing the carrier gas all the heavier hydrocarbons can be eluted and determined gravimetrically. The results are tabulated.
B. B. BAUMINGER

3305. **Gas chromatography. II. Application to gas analysis in the industrial laboratory.** O. Horn, U. Schwenk and H. Hachenberg (Farbwerke Hoechst A.-G., Frankfurt/Main, Germany). *BrennstChem.*, 1958, **39** (21–22), 336–346.—The apparatus described has four alternative columns of different chromatographic materials with tap connections whereby each in turn is used to separate a particular group of gases. Cracking gas is carried in a uniform current of H through a 1·5 to 2·5-metre column containing active carbon and molecular sieve material (5A), to separate and determine the relatively inert gases O, N, CO and CH_4. Argon, if present, is determined with the O, but the two gases can be separated by treatment at $-70°$. If H is to be determined, CO_2 or, for $H < 1\%$, A, is used as carrier (*cf.* Horn *et al.*, *BrennstChem.*, 1957, **38**, 116). Hydrocarbons (C_2 to C_3) are separated by columns of acetonylacetone (15 to 22 metres) or decahydronaphthalene (7·5 metres) on ceramic material. In each case the separation of some pairs is incomplete. In the absence of C_5 hydrocarbons, the separation of diacetylene from vinylacetylene and benzene is best accomplished by a 3-metre column of dioctyl phthalate at 70°, and of C_3H_8 and CO_2 by 2·5 metres of active carbon at 80°. The choice of column in a particular case is facilitated by using a logarithmic scale of the retention times of individual gases with ethane as reference standard. Samples are measured in a fixed-volume by-pass on the inlet line with manometer and vacuum connections, and the ratios of pressure (p) to peak height (h) and peak area for each gas, referred to that of C_2H_2 as unit, are used for quant. calculations. This ratio is not independent of p and is used only for routine analyses of mixtures of similar composition. An example of the analysis of cracking gas having 30 components in proportions 0·1 to 9% is given; it was completed in 3 hr., and 99% of the sample was recovered.
A. R. PEARSON

3306. **Displacement development on ion exchangers. Separation of more than two ions.** C. Cornet, J. Coursier and J. Huré (Dépt. de Métallurg. Chim. Appl., Gif sur Yvette, France). *Anal. Chim. Acta*, 1958, **19** (3), 259–266 (in French).—The earlier work of Coursier and Huré (*Anal. Chim. Acta*, 1958, **18**, 272) on the application of the equilibrium constants of two ions, between solution and resin, to the calculation of the distance of travel necessary for separation, has been extended to systems of more than two ions. The method is applied to the separation of Li^+, Na^+, K^+ and NH_4^+ on Dowex-50 resin, with Ca^{2+} as developer.
T. R. ANDREW

3307. **Electro-chromatography in the study of ions. II.** Hari Gopal Mukerjee (Scottish Church Coll., Calcutta, India). *Z. anal. Chem.*, 1958, **163** (6), 408–411 (in English).—The mobility of inorganic ions and the time - migration distance relationship of ions in different electrolytes are studied. Some fast and slow moving ions in different carrier electrolytes are tabulated.
B. B. BAUMINGER

3308. **An arrangement assuring constancy of pH during column electrophoresis.** L. Auger and G. Boussier (Lab. de Biochimie, Fac. de Méd., 45 rue des Saints-Pères, Paris). *Bull. Soc. Chim. Biol.*, 1958, **40** (9–10), 1379–1381.—In the purification of proteins by electrophoresis on starch columns, lengthy operation times of up to 48 hr. may be required to ensure adequate separation. During this time the pH of the buffer may be altered considerably by electrolysis, resulting in serious disturbances in migration of the proteins. The arrangement described (based on the Mariotte bottle) overcomes this difficulty by allowing the buffer to be changed continuously during electrophoresis, thus maintaining a constant pH. D. W. Moss

3309. **Simple procedure for stabilising the current during paper electrophoresis.** F. Peters, G. Lejeune-Ledant and L. Sanson (Lab. de Chir. Exp., Univ. de Liége, Belgium). *Clin. Chim. Acta*, 1958, **3** (6), 601–603.—The voltage is stepped up from 210 V to 735 V and a resistance approx. 4 times that of the paper strips is placed in series with them. This reduces the effect of alteration in the resistance of the strips during electrophoresis so that the current remains practically constant.
H. F. W. KIRKPATRICK

3310. **Simple method for making starch-gel electrophoretic strips transparent.** S. D. Vesselinovitch (Dept. of Med. and Surgery, Ontario Veterinary College, Guelph, Canada). *Nature*, 1958, **182**, 665.—Direct photometric evaluation of proteins on starch-gel strips is possible. Slice the strip in a specified manner to obtain the central portion and immerse for 2 min. in a satd. soln. of Buffalo black in methanol - H_2O - acetic acid (50:50:1). After washing the strip with the same solvent mixture, without the dye, to remove background staining, boil in 10% acetic acid for 30 sec. to obtain a transparent product. P. D. PARR-RICHARD

3311. **Adaptation of an absorptiometer and a polarograph for automatic recording of the results of paper electrophoresis.** R. Gajzler, Z. Piekarska and T. Stańczuk-Różycka (Inst. Dermatologii i Wenerologii, Warsaw, Poland). *Chem. Anal., Warsaw*, 1958, **3** (2), 159–161.—Use has been made of an absorptiometer (A) and a polarograph (B) connected together so that current generated in the photoelectric cell of A was fed directly to B. The motor

of *B* served simultaneously to move the graph-paper drum and to move the electrophoretic paper in front of the photo-electric cell in *A*. This eliminates the necessity for a voltage stabiliser and a synchronised motor. The apparatus, which is inexpensive in comparison with other devices, is very suitable for the analysis of blood serum and for the continuous analysis of current changes in a photo-electric cell circuit. S. WASILEWSKI

3312. **Apparatus for the quantitative interpretation of paper electrophoresis.** L. Górski and K. Wójcik (I Klinika Chorób Wewnętrznych Akad. Medycznej, Kraków, Poland). *Chem. Anal., Warsaw*, 1958, **3** (2), 137–146.—The apparatus is described, with special reference to protein analysis. The measurement is based on the transmission of monochromatic light through an electrophoretic paper strip treated with liquid paraffin. An extinction *vs.* position of protein fraction diagram is obtained on which planimetric measurements can be made. The total time for an analysis of an electropherogram is 8 to 10 min., 3 to 4 min. for the production of the extinction diagram, and about 5 min. for planimetric measurement.
S. WASILEWSKI

See also Abstract **2828**, Chromatography and ion exchange.

Optical

3313. **New direct-reading attachment for three-metre concave grating spectrograph.** L. O. Eikrem (Engng Dept., Baird Atomic Inc., Cambridge, Mass., U.S.A.). *Appl. Spectroscopy*, 1958, **12** (5), 146–149.—The concn. of 8 different elements can be read simultaneously by means of the attachment described. The 20-inch plate holder of the spectrograph is replaced by an assembly which contains the focal curve, exit slits and photomultiplier tubes. An electronic setting attachment allows the slits to be set rapidly and the stability of the optical alignment is assured by the use of a servo-monitor which automatically and continuously keeps the spectral lines precisely aligned with the exit slits, and is sensitive enough to correct for a 2-μ or 3-μ displacement of the spectrum. K. A. PROCTOR

3314. **Ultra-large spectral apparatus and its application to the investigation of iron and steel.** G. Graue, S. Eckhard and R. Marotz. *Arch. Eisenhüttenw.*, 1958, **29** (10), 619–626.—The presence of a large number of lines in the spectra of heavy metals (*e.g.*, in the determination of Al in steel) introduces difficulties in their analytical evaluation. Complete evaluation requires a high resolving power attainable only with large spectral equipment. A 3·4-metre echelle spectrograph is described in which the principle of crossed dispersion is used. Its applications are discussed and it is pointed out that the apparatus is more suitable for special purposes than for routine analysis.
H. SAWISTOWSKI

3315. **Spectropolarimetry of organic compounds. I. Automatic spectropolarimeter for the u.v. and visible regions.** T. Bürer, M. Kohler and H. H. Günthard (Tech. Hochsch., Zürich). *Helv. Chim. Acta*, 1958, **41** (7), 2216–2219.—The instrument described is a modified Beckman DK-1 spectrophotometer with quartz optics. The standard beam-switching device has been replaced by the Glan-Thompson calcite polariser and analyser and the quartz half-shadow plate of a polarimeter, but here the analyser is geared to a servo-amplifier and the circular half-shadow plate is made of two semicircular pieces of quartz, one laevo- and the other dextro-rotatory and arranged to spin at 12 c.p.s. By means of a phase-sensitive demodulator, the a.c. signal from the standard detector and pre-amplifier of the DK-1 is made to rotate the analyser to the null point, and this movement is traced on the Brown recorder. The over-all range of the instrument is limited by the employment of calcite prisms, but the record does give the optical rotation as a function of wavelength.
E. G. CUMMINS

3316. **Application of the vacuum sliding spark to spectrochemical analysis in the far ultra-violet.** J. Romand, G. Balloffet and B. Vodar (Lab. des Hautes Press., Bellevue, France). *Spectrochim. Acta*, 1958, **13** (1–2), 150–151.—A method has been developed in which the sensitive lines of highly ionised atoms produced in a vacuum spark of a special type (the "sliding vacuum" spark) are used. The spectrum is composed essentially of the lines from the metals of the electrodes, together with a few lines from the elements of the insulator. By using pure aluminium electrodes it is possible to study non-conducting materials such as ceramics, powders, other non-metallic elements (*e.g.*, simple gases), and S and P in steel and aluminium alloys.
B. B. BAUMINGER

3317. **Alternating-beam spectrophotometer.** R. V. Hesketh (Univ. of Glasgow, Scotland). *J. Sci. Instrum.*, 1958, **35** (10), 371–374.—Modifications to a Unicam SP 500 spectrophotometer are described. The light beam from the monochromator is alternated between two paths, one passing through the specimen, and the other through a calibrated attenuator. The attenuator is adjusted to give an absorption equal to that of the specimen, equality being indicated by the absence of an a.c. signal at the photocell on which both beams converge. The instrument covers the wavelength range 2000 to 10,000 A, and in the visible spectrum a change in optical density of 0·0007 can be detected with an illumination intensity of 10^8 quanta per sec. and a response time of 20 sec. G. SKIRROW

3318. **A simple arrangement for the measurement of absorption spectra of aqueous systems at elevated temperatures.** D. W. Grant, D. E. Glanville and G. L. Strachan (A.E.R.E., Harwell, Berks., England). *Spectrochim. Acta*, 1958, **12** (2–3), 109–113.—Details are given of a brass ampoule-holder fitted with nichrome resistance wire for heating. An illustration shows the holder fitted into the cell carriage of a Unicam SP 600 spectrophotometer. Quantitative measurements can be made despite distortions in the optical path arising from the cylindrical nature of the cell and the variation in the refractive index of its contents with temperature. E. G. CUMMINS

3319. **Method of double-beam spectrophotometry in the vacuum ultra-violet.** J. F. James (Dept. of Phys., Univ. of Reading, England). *J. Sci. Instrum.*, 1959, **36** (4), 188–190.—The apparatus permits the measurement of absorption coefficients in the vacuum ultra-violet when the radiation source is unsteady. The beam emerging from a monochromator is divided and one part is passed through an absorption cell. By means of two photomultipliers the intensity ratios of the two beams are compared. An accuracy of 1% at wavelengths down to 800 A can be obtained.
G. SKIRROW

3320. **Analytical applications of far-infra-red spectra. I. Historical review, apparatus and technique.** F. F. Bentley, E. F. Wolfarth, N. E. Srp and W. R. Powell (Wright Air Development Center, Ohio, U.S.A.). *Spectrochim. Acta*, 1958, **13** (1–2), 1–30.—Previous work in the far i.r. region is reviewed and the apparatus and techniques used in obtaining spectra in the caesium bromide region are discussed. The spectra of greases, high polymers and samples which are corrosive to caesium bromide plates can be obtained by using cells of polyethylene and KRS-5 (thallium bromide - iodide) to support the materials. Solvents such as hexane, *cyclo*-hexane, 2:4:4-trimethylpentane, liquid paraffin, benzene, thiophen, furan and dioxan have large windows in the 15 to 35-μ region, and the i.r. spectra of these and a list of more commonly used organic solvents are given. (509 references.)

B. B. Bauminger

3321. **Recent advances in infra-red instrumentation.** S. F. D. Orr (Perkin-Elmer A.G., London). *Spectrochim. Acta*, 1959, **14**, 86–96.—One of the new, cheaper, infra-red instruments, occupying only 27½ in. × 17 in. of bench space, has relatively few adjustments to be made and presents a spectrum recorded between 2·5 and 15 μ on an 8½ in. × 11 in. chart in 12 min. The signal-to-noise ratio is > 100 and the resolution is 0·05 μ throughout. The availability of good cheap gratings has further advanced technique, but short-wavelength cut-off filters have shortcomings which limit the range over which they give reasonable resolution. Spectral impurity is also a drawback beyond 4 μ. These two limitations mean that the filter - grating combination can never replace the present prism. The best filter - grating combination needs two gratings and a succession of about 8 filters to cover the 2·5 to 15-μ range. The resolution of prism instruments (say 5 cm^{-1}) is no longer considered adequate for most chemical purposes. It has been shown that a spectral slit width of 1 cm^{-1} is necessary in future instruments that aim to measure absorbance to within a few per cent. of the true value. Prism - grating instruments that give very high resolution are mentioned. In one of these a Fabry - Perot interferometer was used to attain an accuracy of 0·01 cm^{-1} in wave-number determinations. It is shown that with the technique of scale expansion in which 10% of the scale is shown on the full chart width, the noise level is no more discernible than with single-beam recording. Various optical wedges are introduced into the reference beam to expand the scale required. Energy increase required with scale-expansion sensitivity-increase is less than that required with the sensitivity increase gained by the use of thicker samples. A cell thickness chosen to give an absorbance of $1/e$ will ensure the greatest sensitivity to concentration change in differential analysis. Among the microspectroscopic techniques outlined, mention is made of the reflecting objective prisms now commercially available. Drilled rock-salt plates and KBr discs (1 mm × 3 mm) are among the devices used to obtain spectra on < 10 μg of sample. The need for an internal standard for the correlation of results from one instrument to another is stressed.

E. G. Cummins

3322. **Improved technique for preparing samples of organic solids for infra-red analysis.** R. G. Devaney and A. L. Thompson (Tennessee Eastman Co., Kingsport, U.S.A.). *Appl. Spectroscopy*, 1958, **12** (5), 154–155.—Hard organic solids that are ordinarily difficult to powder for preparing potassium bromide discs may be satisfactorily reduced to a fine powder by abrasion with an inexpensive diamond-surfaced stainless-steel spatula—"Sepi Diachrom Spatula." The 200-mesh unit has proved satisfactory. No contamination with diamond dust has been observed in samples prepared in this way, but the spectra should be examined for possible contamination.

K. A. Proctor

3323. **A method of mounting micro samples for infra-red spectrometry.** D. A. Clark and A. P. Boer (Dept. of Biochem., Texas Univ., U.S.A.). *Spectrochim. Acta*, 1958, **12** (2–3), 276–277.—A method of sample mounting designed for the Perkin-Elmer Model 13 i.r. spectrophotometer with a Model 38A microscope, but having general application, is described. The sample is prepared as a window in a sheet of aluminium foil and mounted at the focal point of the microscope, with the foil as support. The hole is prepared and a third of the foil folded flat underneath it to retain the sample (5 μg), which can be added with or without powdered KBr. The opposite third of the foil is folded flat on top of the sample to enclose it while it is pressed between flat plates in a good vice. When the foil is unfolded the sample is ready for use.

E. G. Cummins

3324. **Difference spectroscopy in the near infra-red.** H. A. Willis and R. G. J. Miller (I.C.I. Ltd., Plastics Div., Welwyn, England). *Spectrochim. Acta*, 1959, **14**, 119–126.—The extra energy available in this region has encouraged experimentation involving greater sample thicknesses. Since the near i.r. is relatively insensitive to changes in proportion of chemically similar, major constituents of mixtures, the minor constituent which does differ chemically will be detectable without elaborate compensating mixtures having to be made. For example, the contaminant of ester copolymers of methyl methacrylate can be measured with the system used for poly(methyl methacrylate). Movable wedges mounted to form a block of rectangular cross-section make a device of variable thickness adaptable to suit sample thicknesses between 0·25 and 1 in. The $>C=CH_2$ absorption has been measured at 1·65 μ after the saturated C–H group absorption at 1·7 μ has been cancelled out.

E. G. Cummins

3325. **Variation of infra-red vibration frequencies with solvents.** A. D. E. Pullin (Chem. Dept., The Queen's Univ., Belfast, N. Ireland). *Spectrochim. Acta*, 1958, **13** (1–2), 125–138.—The change of frequency of a solute band with change in the dielectric constant of the solvent, considered as a continuous dielectric medium, is discussed from the standpoint of dielectric theory, and the effects of several approximations usually necessary in this type of mathematical treatment are considered. The approximation used is that of a point dipole within a spherical cavity in a continuous dielectric medium. The treatment is extended to polyatomic solutes. The lowering of the frequency of the carbonyl vibration of acetone by various solvents, *e.g.*, hexane, decahydronaphthalene, tetrachloroethylene, carbon tetrachloride, carbon disulphide, benzene and dioxan, is tabulated.

B. B. Bauminger

3326. **Polyethylene capillary tubes as micro liquid-cells for infra-red spectroscopy.** W. S. Molnar and V. A. Yarborough (Devel. Dept., Union Carbide Chemicals Co., S. Charleston, W. Va., U.S.A.). *Appl. Spectroscopy*, 1958, **12** (5), 143–145.—In the simple technique described, the minute liquid sample is enclosed in a micro capillary-tube of polyethylene and the spectrum recorded with a commercial i.r. microspectrometer. A modified cold-trap, used with the micro-capillaries for sampling eluates from gas chromatography, is described. K. A. PROCTOR

3327. **Method for elimination of interference fringes in spectra of thin films.** C. Lutinski (The Perkin-Elmer Corp., Norwalk, Conn., U.S.A.). *Anal. Chem.*, 1958, **30** (12), 2071–2072.—By coating a thin film with liquid paraffin and pressing it against a thick rock-salt window, an effective thick sample can be obtained which has minimum surface reflection at the interface, and therefore no observable interference fringe pattern. If the C–H region is of interest, some non-hydrocarbon liquid such as perfluorokerosine can be used instead of liquid paraffin. K. A. PROCTOR

3328. **Elementary analysis by X-ray fluorescence spectrography. I.** C. Raimbault and G. Baron (Inst. Français du Pétrole). *Rev. Inst. Franç. Pétrole*, 1958, **13** (6), 1253–1264.—The theoretical basis of secondary X-ray spectrography is discussed. The method is limited to the detection of elements of atomic number > 19. G. BURGER

3329. **Proportional counters in X-ray spectrochemical analysis.** T. Mulvey and A. J. Campbell (Res. Lab., A.E.I. Ltd., Aldermaston, Berks., England). *Brit. J. Appl. Phys.*, 1958, **9** (10), 406–410.—Analysis for elements lighter than K is discussed and theoretical considerations are reviewed. Statistical curves illustrated would enable alloys of Mg and Si, for example, to be assayed, provided that promised small improvements in proportional counters are successfully achieved. E. G. CUMMINS

3330. **Direct recording of fluorescence excitation spectra.** C. A. Parker (Admiralty Materials Lab., Holton Heath, Poole, Dorset, England). *Nature*, 1958, **182**, 1002–1004.—In the apparatus described, a small proportion of the exciting light is focused on to the front surface of a fluorescent screen which comprises a silica cell containing a suitable fluorescent soln. The fluorescence is viewed through the back of this cell by a photomultiplier (P_1). At the same time a suitable band of fluorescent light from the sample is selected by a monochromator (M_2) and received by a second photomultiplier (P_2). After amplification, the outputs from the two multipliers are passed to a ratio recorder. The frequency drum of the monochromator (M_1) which provides the exciting light is motor-driven, and as the frequency is varied the slits are adjusted so as to maintain the output of P_1 approx. constant (within a factor of 2). If the contents of the monitoring cell are carefully chosen, the output of P_1 can be made proportional to the quantum output of M_1 over a wide frequency range, so that the true fluorescence excitation spectrum of the sample is recorded. Comparisons are given of fluorescence excitation and u.v. absorption spectra. K. A. PROCTOR

3331. **Comparative tests on some instruments used in absorptiometric methods of analysis.** W. E. Clarke (B.C.I.R.A., Alvechurch, Birmingham, England). *J. Res. Brit. Cast Iron Ass.*, 1958, **7** (9), 360–390.—Tests are described for determining the most satisfactory conditions for the Hilger Uvispek spectrophotometer with quartz prism and tungsten-filament light source, the EEL absorptiometer with tungsten-filament light source and Ilford spectrum filters, and the Hilger Spekker absorptiometer with mercury-vapour source and Ilford spectrum filter, in analysis of cast iron; to compare prism- and filter-type instruments and single and double photocell absorptiometers; and to assess the claims of the mercury-vapour lamp as compared with the tungsten-filament lamp in filter absorptiometers. All three instruments were found satisfactory for routine work with the exception of mercury-vapour lamps for Ni, Mo and Ce. The best calibration graphs were obtained with the Uvispek instrument. Recommended conditions are given for Mn, Ni, Cu, Mo, Cr, Ti, V, Ce, Bi, Co and Si. C. H. COWPER-COLES

3332. **Some experiments in precision colorimetry.** Tsunenobu Shigematsu and Masayuki Tabushi (Shigematsu Lab., Kyoto Univ., Kyoto, Japan). *Bull. Inst. Chem. Res. Kyoto Univ.*, 1958, **36** (6), 127–133 (in English).—A study has been made of the accuracy of spectrophotometric determinations by the differential and trace analysis methods, as compared with the conventional method. In the differential method, a soln. of known concn. is used as the reference soln. of zero absorbance. It was found to be more accurate than the conventional method at all concn., and as accurate as gravimetric or volumetric procedures for soln. of high absorbance. In the trace analysis method, the reagent blank is used as zero-absorbance reference, and a soln. of known concn. as the zero-transmittance reference. It is accurate at low concn., but not more so than the conventional method. H. M.

3333. **Development of a commercial automatic colorimetric titrator.** J. M. Thoburn, C. M. Jankowski and M. S. Reynolds (Res. Lab., Central Scientific Co., Chicago 13, Ill., U.S.A.). *Anal. Chem.*, 1959, **31** (1), 124–128.—In the apparatus described, the titration is monitored by a light beam passing through the titration beaker and at the end-point the change in transmittance causes a meter relay to close, which in turn stops delivery of the reagent. Results on most titrations have standard deviations of 0·3% or better for the delivery of 3·5 ml of titrant. K. A. PROCTOR

3334. **Construction and use of a simple reflectometer for the determination of haemoglobin oxygen saturation in blood.** H. E. Refsum and B. Hisdal (Univ. Inst. for Respiratory Physiol., Oslo City Hosp., Norway). *Scand. J. Clin. Lab. Invest.*, 1958, **10** (4), 439–443.—The apparatus and its use are fully described. By using two photocells and a calibrated light shutter, fluctuations in the lamp voltage and uneven illumination of the photocells are minimised, thus giving more stable readings. The instrument is so designed that mains current can be used without stabilisation. The method of calculation eliminates errors due to variations in the sensitivity of the apparatus and in the haemoglobin concn. of the samples; 1·5 to 2·0 ml of blood

is sufficient for a determination. The results compare favourably with those by spectrophotometric methods. D. B. PALMER

3335. Gas analysis by optical interferometry. D. N. Hanson and A. Maimoni (Radiation Lab., Univ. of California, Berkeley, U.S.A.). *Anal. Chem.*, 1959, **31** (1), 77–82.—The optical interferometer is an accurate and reliable instrument for the analysis of binary mixtures of gases. The theory and techniques for the analysis of hydrogen - nitrogen and deuterium - nitrogen mixtures are given. The accuracy is in the order of 0·1 to 0·5%. K. A. PROCTOR

Thermal

3336. Boiling-point apparatus. P. G. Lykos (Illinois Inst. of Technology, Chicago 16). *J. Chem. Educ.*, 1958, **35** (11), 565.—Cottrell's principle of spraying the thermometer with boiling liquid is used. A distinctive feature of the apparatus is the vapour lift which consists of an inner tube with several jets pointing inward near the top and a small drainhole in the bottom which is attached by a ring seal just above the jets to the outer tube the flat bottom of which completes the pump proper. The vapour - liquid mixture moves up through the annular region between the two concentric cylinders. O. M. WHITTON

3337. Thermal electro-analysis. H. F. Holmes and M. J. Joncich (Dept. of Chem., Univ. of Tennessee, Knoxville, U.S.A.). *Anal. Chem.*, 1959, **31** (1), 28–32.—The technique described is based on differential thermal measurements between the anode and cathode, and between these electrodes and the soln., during electrolysis. For a soln. of $CuSO_4$ in the range 0·02 to 0·20 *M*, a relationship between the temp. difference and the concn. of the soln. was established. Concn. can be determined with an accuracy of 1% by this method which has possible applications in the electroplating industry. K. A. PROCTOR

Electrical

3338. Mechanism of electrolytic deposition and dissolution of metals. H. Gerischer (Max Planck Inst. für Metallforschung, Stuttgart, Germany). *Anal. Chem.*, 1959, **31** (1), 33–39.—A review is presented of theoretical principles and recent developments in the investigation of relatively simple electrode reactions, with special attention to fast reactions. Data on complex-ion discharge and the formation or dissolution of solid metals are recorded. K. A. PROCTOR

3339. Practical apparatus for the control of lead - tin alloy plating baths. R. L. Garrett (TWA Overhaul Base, Parkville, Mo., U.S.A.). *Plating*, 1958, **45** (11), 1139–1141.—The determination of the composition of lead - tin alloy plating baths is best effected electrolytically (*cf.* DuRose and Hutchinson, *Ibid.*, 1953, **40**, 470, 630). The design of the cathode is important since it must present a constant area to the soln. A silver-plated brass cathode is described which is shaped like a cylindrical bulb. The surface exposed to the soln. is limited by a mask of acrylic plastic. Both the cathode and anode are held in a plastic fixture from which the cathode may be withdrawn at will. For the determination, two cells in series are used. One, which acts as a coulometer, contains a $Pb(BF_4)_2$ - HBF_4 soln. and uses a pure lead cathode; the other, which contains the sample, uses a lead - tin (93:7) anode. From the measurements, the Sn (%) and the plating time for a given thickness at a given current density are calculated. J. H. WATON

3340. Chloride ion analyser. R. L. Seidel (Knolls Atomic Power Lab., Schenectady, N.Y.). *U.S. Atomic Energy Comm.*, Rep. KAPL–M–SMS–93, Sept. 10, 1958, 35 pp.—A new Beckman silver - silver chloride pressed pellet electrode has been employed in static and flowing systems to measure concn. of Cl^- in the range of 0·1 to 100 p.p.m. The concn. has been measured within ± 0·1 p.p.m. in a quiescent system, and within ± 0·2 p.p.m. at flow rates up to 3 gal. per hr. in a loop containing water from a cooling system contaminated with 0 to 45 p.p.m. of Cl^-. Calibration curves have been prepared for the new electrode used in combination with a non-mercury-containing reference electrode and a model H-2 pH meter at temp. ranging from 10° to 50°. Test results indicate that the cell can be used to monitor the Cl^- content of condensate from steam generators, and in laboratories for a quick determination of Cl^-. NUCL. SCI. ABSTR.

3341. Potentiometric titrations with an attackable electrode. The bimetallic electrode pair platinum - tungsten. I. M. Kolthoff and E. R. Nightingale, jun. (Sch. of Chem., Univ. of Minnesota, Minneapolis, U.S.A.). *Anal. Chim. Acta*, 1958, **19** (6), 593–600.—The potential of a redox system at an attackable electrode (oxidised before evolution of O) depends on the current - potential curve of the electrode in the electrolyte, the concn. of the active ions, the degree of reversibility of the reaction, the true oxidation potential of the system, and the rate of attack of the electrode. These factors are considered for the platinum - tungsten electrode pair in the titration of Fe^{3+} with Ce^{4+}. T. R. ANDREW

3342. The palladium electrode in aqueous and non-aqueous titrimetry. J. T. Stock, W. C. Purdy and T. R. Williams (Dept. of Chem., Univ. of Connecticut, Storrs, U.S.A.). *Anal. Chim. Acta*, 1959, **20** (1), 73–78.—The palladium electrode developed by Schwing and Rogers (*cf. Anal. Abstr.*, 1957, **4**, 1088) has been modified to permit the easy interchange of palladium diaphragms. An improved electrode with a vertical palladium diaphragm and rapid constant-speed stirring is also described. This design allows the escape of hydrogen bubbles and eliminates the buckling which occurs with tightly held palladium discs. The new electrode works best at low c.d. and examples are given of several titrations of weak and strong acids and organic bases in H_2O, 95% ethanol and dimethylformamide. The results are satisfactory except when pptn. occurs. W. T. CARTER

3343. The choice of bimetallic electrodes for the potentiometric titration in argentimetry. Kozo Ueno and Tatsuhei Tachikawa (Muroran Univ. of Engng, Hokkaido). *Japan Analyst*, 1958, **7** (12), 757–761.—The change in potential during the titration of halide ions was examined with four pairs of electrodes, *i.e.*, nickel, silicon, antimony or tungsten *vs.* platinum (anode). An appreciable change in potential difference (*e.g.*, ≃40 mV. for 0·1 *N* soln., 5 to 20 mV for 0·01 *N* soln.) was observed, although the precise shapes of the curves differed. The end-point coincides with that obtained by the usual indicator. K. SAITO

3344. Voltammetric studies at the graphite electrode in quiet solutions. J. B. Morris and J. M. Schempf (Chem. Dept., Pennsylvania State Univ., University Park, U.S.A.). *Anal. Chem.*, 1959, **31** (2), 286–291.—The electro-reduction of Ag^{I} and Cu^{II} has been studied in various media with an impregnated graphite electrode specially prepared to give a reproducible, renewable surface without special electrical treatment before use in voltammetry. Such an electrode exhibits extended useful polarisation ranges compared with platinum, and studies on the ferri - ferrocyanide system indicate that reaction-reversibility is established at the electrode surface. K. A. PROCTOR

3345. Device for magnetic stirring in electrometric titration. P. Hansen (Central Lab., Odense City and County Hosp., Denmark). *Scand. J. Clin. Lab. Invest.*, 1958, **10** (3), 345.—A plastic stirrer, in the shape of an inverted "T," with a piece of iron enclosed in the lower horizontal bar, is suspended from a bearing in a Perspex plate. The plate, which also carries the electrodes and the tip of the burette, fits over the titration vessel; thus the whole assembly including the stirrer can readily be transferred from vessel to vessel.

D. W. MOSS

3346. Automatic continuous coulometric titration. IV. Continuous coulometric titration with electrolytically generated ferrous ions. Takeo Takahashi and Hiroshi Sakurai (Inst. Ind. Sci., Tokyo Univ., Yayoicho, Chiba). *Japan Analyst*, 1958, **7** (10), 631–636.—The potential difference between the platinum and calomel electrodes in the reaction cell is compensated by a reference potential, which is amplified and applied to the electrolytic reduction of $Fe_2(SO_4)_3$ in 2 *M* H_2SO_4, to produce the amount of Fe^{2+} required to keep the redox reaction in the cell in an equilibrium state. Both the sample (containing $KMnO_4$, $K_2Cr_2O_7$ or Cl) and the Fe^{2+} soln. are introduced into the reaction cell at a const. rate (*e.g.*, 0·16 ml per sec.). The amount of the oxidising agent is indicated by the current used in the electrolysis of Fe^{3+}, which is automatically recorded with time. For the determination of the reference potential, a curve of electrolytic current *vs.* voltage between the platinum and calomel electrodes is used. K. SAITO

3347. Automatic coulometric titrator for the Karl Fischer determination of water. Application of the Leeds and Northrup alternating-current operated pH indicator to the control of titrations. M. T. Kelley, R. W. Stelzner, W. R. Laing and D. J. Fisher (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1959, **31** (2), 220–221.—In the coulometric titration arrangement described and illustrated, an automatic cut-off is attached to the pH indicator, so that the whole instrument can be used to control either pH or millivolts, including dead-stop measurements. The titrator is designed specially for the constant-current coulometric determination of H_2O by the method of Meyer and Boyd (*cf. Anal. Chem.*, 1959, **31**, 215). For this purpose the instrument has a range of 5 sec. at 50 mA to 100 sec. at 300 mA, corresponding to 0·02 and 3 mg of H_2O, respectively.

W. J. BAKER

3348. Background corrections in controlled-potential coulometric analysis. L. Meites and S. A. Moros (Dept. of Chem., Polytechnic Inst. of Brooklyn, N.Y., U.S.A.). *Anal. Chem.*, 1959, **31** (1), 23–28.—Experimental data are given for the properties of "charged," "faradaic," "kinetic" and "induced" backgrounds in controlled-potential coulometric analysis, and the application of corrections for them in practical analysis is discussed.

K. A. PROCTOR

3349. Amperometric titrations with solid indicator-electrodes. J. T. Stock (Dept. of Chem., Univ. of Connecticut, Storrs, U.S.A.). *Microchem. J.*, 1958, **2** (2), 229–244.—Amperometric titrations, particularly of metals, performed at all forms of solid electrodes are surveyed. Developments since a previous review (*Metallurgia, Mnchr*, 1947, **36**, 51) are covered. (118 references.)

D. F. PHILLIPS

3350. Redoxokinetic titration—a new electroanalytical technique. K. S. G. Doss, U. H. Narayanan and K. Sundararajan (Central Electrochem. Res. Inst., Ramanad Distr., Karaikudi, India). *Analyst*, 1958, **83**, 696–698.—The rectifying property of reversible electrodes (Doss and Agarwal, *J. Sci. Ind. Res., B, India*, 1950, **9**, 280), designated the "redoxokinetic effect" since the rectification is dependent on the kinetics of oxidation - reduction reactions at the electrodes, is applied to the determination of end-points in redox titrations. Acidified 0·02 *N* $(NH_4)_2SO_4.FeSO_4$ is titrated with $KMnO_4$ soln. After each addition of $KMnO_4$ the redoxokinetic potential is measured with a galvanometer at a fixed a.c. potential. The titration may also be made under constant current conditions, *i.e.*, without re-adjustment of the a.c. potential to a particular value after each addition of titrant, and this simplifies the procedure. The apparatus is described and results with both procedures are shown graphically. The end-point can be determined with an error of $\pm 0{\cdot}4\%$. A. O. JONES

3351. Radiometric titrations with Complexones. T. Braun, I. Maxim and I. Galateanu (Inst. of Atomic Physics, Casuta Postală Nr. 35, Bucharest). *Nature*, 1958, **182**, 936–937.—A cation is titrated with a complexing agent in the presence of a ppt. containing a radioactive cation, which acts as an indicator. The radioactivity of the soln. remains constant at a low level until the end-point, and then increases linearly with the vol. of titrant added. The procedure is exemplified by the titration of 0·01 *M* Cu^{2+} at pH 9 with 0·01 *M* EDTA, with $^{110}AgIO_3$ as indicator; after the end-point the $^{110}AgIO_3$ dissolves in the excess of EDTA.

A. R. ROGERS

3352. Gamma-ray spectroscopy applied to radioactivation analysis. I. Introduction and nuclear data. L. Salmon (A.E.R.E., Harwell, Berks., England). A.E.R.E. Report C/R 2377 (1), 1959, 92 pp.—The equipment required is considered briefly. Data, including half-lives, energies and relative intensities of the γ-rays emitted, are tabulated for nuclides with half-lives of 4·5 min. or longer that are produced by (n, γ) reactions. Spectra are illustrated for many such nuclides and for some fission products and naturally radioactive materials. G. J. HUNTER

3353. Chemical analysis by means of nuclear magnetic resonance. W. G. Proctor (Varian Ass. Wassenaar, Netherlands). *Z. anal. Chem.*, 1958, **164** (1), 127–134 (in English).—The phenomenon and use of nuclear magnetic resonance are explained and illustrated by a number of spectra.

B. B. BAUMINGER

3354. The practice and application of nuclear resonance spectroscopy. R. E. Richards (Phys. Chem. Lab., Oxford). *Spectrochim. Acta*, 1959, **14**, 5–16.—The basic formulae of this technique, which involves a study of the absorption of radio-frequency energy by nuclei in a strong uniform magnetic field—$\nu \equiv \gamma H/2\pi \equiv \mu H/Ih$, where the nuclear resonance signal occurs at frequency ν, and γ is the magnetogyric ratio for the nucleus, μ is its nuclear magnetic moment, I the nuclear spin quantum number, h is Planck's constant and H is the magnetic field experienced by the nucleus, and—$H \equiv H_0 (1 - \sigma)$, where H_0 is the applied magnetic field and σ is a variable, due to surrounding electron clouds, depending on the compound in which the nucleus is situated ('magnetic shielding' giving rise to 'chemical shifts'), are discussed. The theory of chemical shifts and spin-spin interaction, and such qualitative applications as structural diagnosis and the study of chemical equilibria, are reviewed. A block diagram of a simple apparatus using < 1 kW power, incorporating a permanent magnet giving 7000 G, from which information on hydrogen or fluorine resonance has been obtained, is included. Electromagnets can create a field of 15,000 G but elaborate means are needed to keep it stable.
E. G. CUMMINS

3355. A new method for analysing spectra in high-resolution N.M.R. spectroscopy. H. Primas (Swiss Federal Inst. of Technol.). *Spectrochim. Acta*, 1959, **14**, 17–23.—An outline of the calculation of resonance frequencies and intensities by classical methods such as diagonalisation of the interaction Hamiltonian, cumbersome for complicated molecules, and the moment method which has been applied to the analysis of spin-spin multiplets in high-resolution work, is followed by the explanation of a correlation function of the spectrum, from which all known methods of analysis can be deduced; this has been derived from a mathematical investigation of spin-spin coupling, the full development of which is to be published. Approximations to be employed in both perturbative and non-perturbative expansions of the correlation function are explained, and a non-perturbative cluster expansion, which neglects the higher-order many-particle interactions, is included.
E. G. CUMMINS

3356. Analysis of complex systems by the method of β-ray reflection. S. N. Kryukov, B. S. Bokshteĭn, T. I. Degal'tseva and A. A. Zhukhovitskiĭ (I. V. Stalin Moscow Inst. of Steel). *Zavod. Lab.*, 1958, **24** (11), 1305–1308.—The analysis of mixtures of Fe, Mo and W and the determination of Fe in ores are studied. The source of radiation is the equilibrium mixture of ^{90}Sr and ^{90}Y, and the reflected radiation is measured after passage through filters (aluminium) of various thicknesses. With Fe-Mo-W mixtures and the use of thick filters, samples of the same mean mol. wt. give the same intensities whilst, with the use of thin filters, samples of the same mol. wt. give intensities that depend on the content of one component (Fe). Measurements with two filters and comparison with standards permit the contents of all three components to be determined. When the contents are approximately the same, the mean error is $\simeq$ 0·5%.
G. S. SMITH

3357. Time-of-flight mass spectrometry and gas-liquid partition chromatography. R. S. Gohlke (Dow Chem. Co., Midland, Mich., U.S.A.). *Anal. Chem.*, 1959, **31** (4), 535–541.—The direct combination of a time-of-flight mass spectometer, which scans a mass range from $m/e = 1$ to $m/e = 6000$ at the rate of 2000 times per sec., with a gas-liquid partition chromatography apparatus produces an instrument capable of rapidly and completely characterising organic chemical mixtures boiling at < 350°.
K. A. PROCTOR

3358. Interference effects in the mass-spectrometer ion-source. R. Taubert (Phys.-Tech. Bundesanstalt, Braunschweig, Germany). *Z. anal. Chem.*, 1958, **164** (1), 164–181.—The short-term interference (measured in sec.) is a space-charge effect and can be eliminated by proper construction and adjustment of the ion-source. The long-term effect (measured in min.) is caused by a change in the cathode properties. This effect can be eliminated transiently by pre-treatment with but-1-ene.
B. B. BAUMINGER

3359. Method for the correction of inverse matrix in chemical analyses with the mass spectrometer. N. Calderón L. and H. Ehrhardt (Phys. Inst., Univ. Bonn, Germany). *Z. anal. Chem.*, 1958, **164** (1), 30–36.—A convergent approximation procedure is described for the correction of the inverse matrix in mass spectrometry which can be accomplished with a small computer in a few hours.
B. B. BAUMINGER

3360. The automatic control of mass-spectrometric gas analysis. J. van Katwijk (Koninklijke/Shell-lab., Amsterdam, Holland). *Z. anal. Chem.*, 1958, **164** (1), 73–80.—Procedure and apparatus for the analysis of gas mixtures using an "analogue-to-digital converter" (*cf.* Dudenbostel and Priestley, *Anal. Chem.*, 1954, **26**, 1275) and an electronic computer are described. This method is quicker than the normal gas-spectrometric analysis and photographic registration of the peaks, and is shown to be of equal accuracy.
B. B. BAUMINGER

3361. Mass-spectrometric analysis. Molecular rearrangements. F. W. McLafferty (Eastern Res. Lab., The Dow Chemical Co., Framingham, Mass., U.S.A.). *Anal. Chem.*, 1959, **31** (1), 82–87.—A classification of molecular rearrangements involved in structural determinations by mass spectrometry is presented.
K. A. PROCTOR

3362. Mass spectrometer ion source for solid samples. B. R. F. Kendall (Dept. of Physics, Univ. of W. Australia, Nedlands). *Rev. Sci. Instrum.*, 1958, **29** (12), 1089–1092.—The sample is placed on a single filament ionisation source at a point where the temp. is high enough for evaporation but too low for significant surface ionisation. Constructional details are given and operating conditions discussed.
G. SKIRROW

3363. Comparisons of spectra from different types of mass spectrometers. V. J. Caldecourt (Spectroscopy Lab., Dow Chemical Co., Midland, Mich., U.S.A.). *Appl. Spectroscopy*, 1958, **12** (6), 167–171.—A comparison of the cracking patterns of various compounds taken on different types of mass spectrometer shows large differences. Mass discrimination is a large factor, which is to be expected, particularly when comparing 180° with 60° or 90° instruments. Variations due to the nature of the compounds are also evident.
K. A. PROCTOR

3364. Derivation of mass spectra of individual compounds from the spectra of mixtures. S. Meyerson [Res. Dept., Standard Oil Co. (Indiana), Whiting, U.S.A.]. *Anal. Chem.*, 1959, **31** (2), 174–175.—The method described is based on the analysis of two different mixtures of the two components of a mixture under study. By deducting all the peak heights of one spectrum (multiplied by an appropriate factor) from the corresponding peak heights of the other, a difference spectrum for either component can be obtained. In favourable cases the method can be extended to three- and four-component mixtures, and is particularly useful for correcting reference spectra for unknown impurities.
K. A. PROCTOR

3365. Directional scintillation counter. M. A. Thompson (Univ. of Auckland, N.Z.). *Rev. Sci. Instrum.*, 1958, **29** (12), 1149.—The counter consists of a bundle of Pyrex-glass capillary tubes filled with the phosphor. The light generated within the phosphor is collected by two photomultipliers, one on each side of the bundle, and the coincidences between the resulting pulses are recorded. The counters are tested by comparing counting rates due to a neutron flux in the axial and broadside directions.
G. SKIRROW

3366. Thermocouple method for measuring relative humidity in the range 95 to 100%. J. L. Monteith and P. C. Owen (Rothamstead Exp. Sta., Harpenden, Herts, England). *J. Sci. Instrum.*, 1958, **35** (12), 443–446.—At high relative humidities a current of 30 mA through a 38 s.w.g. chromel-*p*/constantan thermocouple produces sufficient Peltier cooling to form a film of water on the junction, which may then be used as the wet-bulb of a psychrometer. The wet-bulb depression can be read to 0·001° and in the range 97 to 100%, changes of 0·01% r.h. can be detected.
G. SKIRROW

ERRATA

June (1956) issue, abstract 1739, lines 1 and 4. *For* "3-hydroxy-1:3-diphenyltriazine" *read* "3-hydroxy-1:3-diphenyltriazen."

March (1957) issue, abstract 821, lines 1 and 7. *For* "3-hydroxy-1:3-diphenyltriazine" *read* "3-hydroxy-1:3-diphenyltriazen." Abstract 821, line 3 and fourteenth line from bottom. *For* "3-hydroxy-1-*p*-chlorophenyl-3-phenyltriazine" *read* 1-*p*-chlorophenyl-3-hydroxy-3-phenyltriazen."

September (1957) issue, abstract 2989, line 1. *For* "3-hydroxy-1-*p*-sulphophenyl-3-phenyltriazine" *read* "3-hydroxy-3-phenyl-1-*p*-sulphophenyltriazen."

These errata occur in the original papers.

January (1959) issue, abstract 34, line 13. *For* "limit of dilution is in 5×10^7" *read* "limit of dilution is 1 in 5×10^7."

January (1959) issue, abstract 142, line 3. *For* "Araf'eva" *read* "Aref'eva."

January (1959) issue, abstract 147, third line from bottom. *For* "Zn" *read* "Zr."

January (1959) issue, abstract 151, line 3. *For* "J. Čepalák" *read* "J. Čepelák."

March (1959) issue, abstract 883, line 3. *For* "E. P. Nikolaeva" *read* "E. R. Nikolaeva."

April (1959) issue, abstract 1350, line 12. *For* "primary" *read* "tertiary."

May (1959) issue, abstract 1657, line 2. *For* "M. V. Kuhn" *read* "V. Kuhn."

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	A
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
constant	const.
corrected	(corr.)
crystalline / crystallised	cryst.
cubic	cu.
current density	c.d
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
ethylenediaminetetra-acetic acid	EDTA
electromotive force	e.m.f.
equivalent	equiv.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_λ^t
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	% (v/v)
per cent. (wt. in vol.)	% (w/v)
per cent. (wt. in wt.)	% (w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	R_F
relative humidity	r.h
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{3+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{2-} PO_4^{3-}, etc., for anions.

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ANALYTICAL ABSTRACTS

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